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FOREWORD

This report describes the research activity carried out in fulfillment of Contract NAS3-4174 during the period from November 15, 1966, through May 14, 1967. The work was conducted under the direction of the Space Power Systems Division, Lewis Research Center, National Aeronautics and Space Administration, with J. A. Milko as Project Manager. Contributors to this report included C. Williams and R. Persons. Previous reports covering work under this contract are

CR-54268 (PWA-2518)
CR-54444 (PWA-2608)
CR-54891 (PWA-2750)
CR-72058 (PWA-2877)
CR-72149 (PWA-2979)

ABSTRACT

The long-term total hemispherical emittance testing of two AISI-310 stainless steel tubes coated with calcium titanate and iron titanate was completed. Both specimens were tested at 1350°F in a vacuum of 1 x 10^{-7} mm Hg or better for 20,000 hours. The calcium-titanate-coated specimen demonstrated an emittance which decreased gradually from 0.91 to 0.88 during the test. The coating remained extremely well bonded to the substrate with no significant changes occurring to the coating. The tested substrate was found to contain considerable quantities of both sigma and carbide phases. The substrate was slightly harder after testing than before, presumably as a result of the precipitation of sigma and carbide phases. No significant interdiffusion of the coating and substrate constituents occurred. The emittance of the iron-titanate-coated specimen remained essentially constant, with only a slight decrease from 0.89 to 0.87 during the test. The coating separated from the substrate at the ends of the specimen and around the black-body holes, but the area affected in the test section was small, and no significant change in the emittance was observed. Testing also resulted in some changes in the coating structure. This consisted of a partial decomposition of the iron titanate into titanium oxide and ferrous oxide. No significant interdiffusion of coating and substrate constituents occurred. The changes in the substrate were identical to those which occurred in the substrate of the calcium-titanate-coated specimen.

A 5,000-hour cycling test of an iron titanate-coated columbium -1 percent zirconium tube was completed. The specimen was cycled between 1500 and 1800°F a total of 3,125 times in a vacuum of 1 x 10^{-7} mm Hg or better. The time to complete one cycle was 96 minutes. The coating demonstrated a stable emittance of about 0.88 and had good adherence to the substrate for the duration of the test. Some changes in the structure of the iron titanate coating occurred, with both iron titanate and titanium oxide phases detected after testing. Limited amounts of iron and titanium and a significant amount of oxygen diffused into the substrate. It was found that partial recrystallization of the substrate resulted from testing. It was also found that the substrate was significantly harder than would be expected for the partially recrystallized alloy. This hardness increase is attributed to the diffusion of iron, titanium, and oxygen into the substrate.

A 1500 to 1900°F cycling test of an iron titanate-coated columbium -1 percent zirconium tube was continued. The specimen had completed 5,000 hours of testing which includes 3,115 cycles. The emittance remained stable at about 0.89. Some cracking occurred at the ends of the tube after about 3500 hours, but no spalling or separation of the coating was observed. The test will be continued to 10,000 hours.

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I. INTRODUCTION

A program is being conducted to determine the suitability of selected high-emittance materials for use as coatings on nuclear space powerplant radiators. The coating materials are being evaluated at elevated temperatures and high vacuum with respect to emittance stability, adherence, and compatibility when applied to AISI-310 stainless steel, columbium-1 percent zirconium, and beryllium substrates.

The work is divided into three phases. One of these involved the evaluation of selected materials coated on beryllium and aged at 800 and 1200°F for periods up to 1000 hours. This phase was completed, and the results were reported in the semiannual progress report submitted in June 1966 (CR-72058, PWA-2877). The second phase involved the evaluation of selected coatings on columbium-1 percent zirconium and AISI-310 stainless steel for periods up to 20,000 hours. This phase has also been completed, and the results for the coated stainless-steel specimens are presented in this report. The third phase of the program consists of two 5000-hour thermal cycling endurance tests on iron-titanate-coated columbium-1 percent zirconium specimens. One of these has been completed, and the second has been extended by an additional 5000 hours.

II. BACKGROUND

Aging tests to determine the emittance stability, adherence, and compatibility of selected materials coated on beryllium plates were completed during the report period ending May 14, 1966. The materials selected for these tests were calcium titanate, iron titanate, and zirconium titanate, and all coatings demonstrated adequate adherence and compatibility with beryllium for periods up to 1000 hours at 1200°F, although the bond strength of the zirconium-titanate coating was somewhat weaker than that of the other coatings. Aging produced no changes in the coatings or substrates other than permanently increasing the emittance of calcium titanate to 0.92. Iron titanate and zirconium titanate both demonstrated stable emittance values of 0.91 at 900°F.

The testing of coated columbium-1 percent zirconium tubes at 1700°F for 10,000 hours was also completed during the report period ending May 14, 1966, and the results indicated that iron-titanate has adequate adherence characteristics and an emittance in the range of 0.88 to 0.84. A small amount of diffusion of coating constituents into the substrate and of zirconium into the coating occurred, as well as diffusion of oxygen into the substrate across both the coated and uncoated surfaces. The second coating material tested on a columbium-1 percent zirconium tube was aluminum oxide aluminum titanate. This material deteriorated during testing.

III. LONG-TERM EMITTANCE TESTING

A. Introduction

The long-term total hemispherical emittance testing of two AISI-310 stainless steel tubes coated with calcium titanate and iron titanate was completed during the report period. These tests were originally scheduled to be terminated after 5000 hours, but in May 1965 the test period was extended to 10,000 hours, and in May 1966 the period was extended to 20,000 hours. Both tests were conducted in vacuums of 1×10^{-7} mm Hg or better at 1350°F with periodic thermal cycling to room temperature. The time required to cool the specimen from 1350°F to 300°F was approximately one minute, and the time required to reheat the specimen to the test temperature was approximately four minutes.

B. Test Results

1. Calcium Titanate on AISI-310 Stainless Steel

With the addition of 755 hours of testing during the current report period, the AISI-310 stainless steel tube with a 4-mil thick coating of calcium titanate completed the scheduled 20,000-hour total hemispherical emittance test in vacuum at 1350°F. The specimen was subjected to a total of 112 thermal cycles from 1350°F to ambient temperature during the test with 9 cycles occurring during this report period. No apparent adverse effects on emittance or adherence of the coating resulted from the cycling. The appearance of the specimen at 1350°F shortly before termination of the test is shown in Figure 1.

The emittance of the specimen was excellent throughout the test. As shown in Figure 2, the emittance measured 0.91 at the beginning of the test, decreased gradually to 0.88 after 13,000 hours, and remained steady at about 0.88 for the final 7,000 hours. The vacuum maintained in the test chamber during the report period was 1×10^{-7} mm Hg or better.

Examination of the specimen following the test revealed a slight darkening of the coating, but no change in texture was apparent, nor was there any indication of cracking or spalling. Attempts to mechanically remove the coating with a silicon-carbide scraping tool indicated that the coating was extremely well bonded to the substrate. Figure 3 shows the specimen after completion of the test.

Semi-quantitative spectrographic analyses were made on the coating both immediately after application and after endurance testing. These results are shown in Table 1.

TABLE 1
Semiquantitative Spectrographic Analysis Results
for Calcium-Titanate Coating on
AISI-310 Stainless Steel Tube

Impurity Content (Weight Percent)

	Fe	Cr	Ni	Al	Mn	Cu	Si	Mg
As Sprayed	0.2*	0.1*	0.07*	-	0.01	<0.01	<0.01	<0.01
After Test	-	0.1	-	0.01	0.5	<0.01	0.05	<0.01

* Presence of these constituents is attributed to small quantities of stainless steel introduced when coating was scraped from substrate.

X-ray diffraction analysis of the coating in the as-sprayed condition and after testing detected calcium titanate as the only crystalline phase present in both cases.

The tested specimen and a sample of the substrate tubing in the as-received condition were sectioned for metallographic examination. The structure found in the as-received sample (Figure 4) is considered to be normal for annealed AISI-310 stainless steel. The microstructure of the tested specimen, as shown in Figure 5, contains substantial amounts of precipitates in an austenitic matrix. Identification of the precipitated phases was attempted by metallographic techniques. The results indicated that the more massive of the two distinguishable phases in the matrix was sigma phase and that the smaller, more finely divided phases were carbides.

The photomicrographs shown in Figure 5 were etched to reveal the precipitates, but the etchant removed material from the interface between the coating and the substrate, making it appear that separation had occurred. The fact that no separation did occur is evident in the unetched photomicrograph shown in Figure 6.

Microhardness traverses were made on the cross section of the stainless steel tube in the as-received condition and on the substrate after testing. Measurements were made with a Reichert microhardness tester using a load of 43 grams. The Reichert microhardness tester is similar to the Tukon tester with the only difference being the shape of the indenter. The tube in the as-received condition had a diamond pyramid hardness of about 200 kg/mm^2, indicating that the material was fully annealed. This hardness was uniform throughout the cross section. The diamond pyramid hardness of the endurance-tested tube was about 245 kg/mm^2, with no significant variations measured throughout the cross section. These results are shown in Table 2.

TABLE 2
Microhardness Traverse Results for Endurance-Tested AISI-310 Stainless Steel Tube Coated With Calcium Titanate

Depth Below Interface (Mils)	Diamond Pyramid Hardness (kg/mm^2) Traverse 1	Traverse 2
0.75	239	244
2.0	244	-
2.5	248	235
4.25	244	235
6.0	239	248
7.5	244	253
9.5	239	239

Note: Total wall thickness equals 10 mils.

In order to obtain more information concerning the changes which occurred in the substrate of the tested specimen and also to determine whether any coating-substrate interaction occurred, electron beam microprobe analyses were conducted on both an untested substrate and the tested substrate. The analyses were performed with a Norelco electron probe microanalyzer.

Initially, electron microprobe and wet chemical analyses were performed on the stainless steel tube in the as-received condition. The results of the two analyses, shown in Table 3, were in reasonable agreement and indicated that the material conformed to the specifications established for the alloy.

The relative concentrations of iron and chromium determined by X-ray line scans across the stainless steel in the as-received condition indicated that the material was homogeneous (Figure 7).

The scan of the tested tube, as shown in Figure 8, reveals a considerable variation in both iron and chrome levels as a function of position within the substrate. The regions which showed higher than average chrome and lower than average iron are believed to be zones in which the electron beam impinged on the massive precipitate.

Quantitative point-count analyses (concentrations of specific elements at specific points in the sample) were performed to determine the composition of the phases formed during the endurance test. The results, shown in Table 4, indicate that the more massive phase falls into the sigma phase composition, as indicated previously in the metallurgical examination section. The austenitic matrix material had a higher iron and nickel content and a lower chromium content than the sigma phase. The over-all composition in the tested specimen was essentially the same as that of the specimen in the as-received condition, indicating no preferential loss of significant quantities of material during testing.

TABLE 3

Composition of AISI-310 Stainless Steel
Tube in As-Received Condition

	Concentration (Weight Percent)			
	Nominal		Wet Chemical	Electron Micro-
	Min	Max	Analysis	Probe Analysis
Carbon	0	0.25	0.08	-
Manganese	0	2.00	1.85	1.0
Silicon	0	0.75	0.64	0.7
Phosphorus	0	0.040	-	<0.1
Sulfur	0	0.030	-	<0.1
Chromium	24.00	26.00	25.19	24.9
Nickel	19.00	22.00	19.50	20.2
Molybdenum	0	0.50	0.20	-
Copper	0	0.50	-	-
Iron	Balance		Balance	51.4

TABLE 4

Composition of AISI-310 Stainless Steel Coated
With Calcium Titanate and Tested
for 20,000 Hours at 1350°F

	Composition (Weight Percent)				
Material	Cr	Ni	Mn	Fe	Si
Sigma Phase	42.1	9.1	0.8	45.4	1.1
Matrix	18.8	21.7	1.7	55.7	0.6
Over-All Composition	24.7	20.4	1.1	51.5	0.8

The substrate was examined for evidence of titanium diffusion by quantitative point-count analysis, and the results are shown in Table 5. As shown, the titanium concentration decreased from 0.07 weight percent at the substrate surface to less than 0.01 weight percent at a depth of 1.5 mils, indicating that only a very limited amount of titanium diffusion occurred during the test.

TABLE 5
Titanium Concentrations in AISI-310 Stainless Steel Coated With Calcium Titanate and Tested for 20,000 Hours at 1350°F

Depth Below Outer Surface (Mils)	Concentration (Weight Percent)
0.25	0.07
0.75	0.02
1.50	<0.01
3.75	<0.01
7.0	<0.01

Note: Total wall thickness equals 10 mils.

The results shown in Table 5 were verified by scanning across the coating-substrate interface and measuring the relative concentrations of titanium and iron. The results (see Figure 9) clearly demonstrate the absence of any significant interdiffusion of these elements.

A vacuum fusion analysis was performed on the substrate in the as-received condition and after testing. The oxygen content in the tube before and after testing was found to be 0.041 and 0.049 weight percent, respectively, which is within the accuracy of the apparatus. The analysis of the tested substrate was performed across the full section thickness after the coating had been mechanically removed and the surface had been cleaned by acid etching. Since there was no significant change in the oxygen content, the oxygen distribution across the substrate was not determined.

In summary, the calcium-titanate-coated AISI-310 stainless steel tube tested for 20,000 hours in vacuum at 1350°F demonstrated a stable emittance of about 0.89 and excellent bonding between the coating and the substrate. Only a very slight amount of interaction between the coating and the substrate occurred. In the substrate, a substantial amount of sigma and carbide phases precipitated, Prolonged exposure at 1350°F would have a tendency toward sigma phase formation as observed after 20,000 hours of testing. The formation of these precipitates in the substrate is not related in any way to the coating. The increase in the substrate hardness observed after testing is believed to be due to the presence of these precipitates, particularly the finely divided phase.

2. Iron Titanate on AISI-310 Stainless Steel

The AISI-310 stainless steel tube with a 4-mil thick coating of iron titanate accrued 1,630 hours of testing during the report period to complete the scheduled 20,000-hour test at 1350°F in vacuum. The specimen was thermally cycled an additional 11 times from 1350°F to ambient temperature during this period, completing a total of 114 cycles for the duration of the test.

The emittance of the specimen was very stable throughout the test with only a slight decrease from 0.89 to 0.87 during the 20,000-hour period (see Figure 10). As shown in Figure 10, the vacuum maintained in the test chamber was about 2×10^{-8} mm Hg.

As reported in the last semiannual progress report, coating separation occurred at the ends of the specimen and around the black-body holes after about 14,500 hours of testing. The progression of the separation is shown in Figure 11. The start of separation at the ends of the specimen was most likely caused by crimping the ends of the tube for electrical connections. The emittance of the specimen was not affected by the separation at the ends of the tube since emittance measurements are made only for the central 1.5 inches of the tube. Separation around the black-body holes, which are in the test section, involved less than two percent of the test section area, and, therefore, had an insignificant effect on the measured emittance. This coating separation is probably cause by mechanical stresses applied to the coating and substrate during machining of the black-body holes or during the reaming process after plasma-spraying of the coating.

The appearance of the specimen at room temperature after completion of the test is shown in Figure 12. The texture and integrity of the coating was unchanged from that observed at the start of the test, and only a slight change in color occurred. The adherence of the coating to the substrate was good except at the ends of the specimen and in the vicinity of the black-body holes.

The coating was subjected to semiquantitative spectrographic analysis before and after testing, and the results are shown in Table 6. The changes which occurred were small and generally were within the accuracy limits of the analysis.

TABLE 6
Semiquantitative Spectrographic Analysis Results for Iron Titanate Coating on AISI-310 Stainless Steel Tube

	Impurity Content (Weight Percent)						
	Al	Mn	Cu	Mg	Cr	Si	Ni
As Sprayed	0.7	0.5	0.01	0.1	0.05	0.3	0.04
After Test	0.5	1.0	<0.01	0.5	0.2	0.5	

X-ray diffraction analysis of the coating indicated that testing did cause some changes in the structure of the coating. In the as-sprayed coating, the only crystalline phase detected was iron titanate. After testing, about 75 percent of the material was iron titanate, about 20 percent was titanium oxide, and the remainder was ferrous oxide.

The endurance-tested coated specimen was sectioned for metallographic analysis. The etched photomicrograph (see Figure 13) shows that the substrate contained precipitates which were identical in appearance to those observed in the calcium-titanate-coated specimen. The slight difference in size and distribution of the precipitates in this substrate from those observed in the calcium titanate-coated substrate is considered to be due to normal variations in the precipitation reaction from section to section. An unetched photomicrograph is shown in Figure 14. As shown, no separation is evident between the coating and the substrate.

Two microhardness traverses were made on a cross section of the endurance-tested tube. The average diamond pyramid hardness was about 210 kg/mm^2, which represents a 10 kg/mm^2 increase from the untested state. There were no significant variations across the tube wall. These results are shown in Table 7.

TABLE 7
Microhardness Traverse Results for Endurance-Tested AISI-310 Stainless Steel Tube Coated With Iron Titanate

Depth Below Interface (Mils)	Diamond Pyramid Hardness (kg/mm^2) Traverse 1	Traverse 2
0.5	211	211
2.0	208	-
2.5	-	219
3.5	215	-
3.75	-	215
5.5	208	-
6.0	-	208
7.0	208	-
7.5	-	211
9.0	211	208

Note: Total wall thickness equals 10 mils.

Electron microprobe analyses were conducted on the tested specimen to identify the precipitates which formed in the austenitic matrix. Quantitative point count analyses for iron and chromium were performed on the more massive precipitate, and the results indicated that this precipitate was sigma phase. The results were essentially identical to those obtained from the calcium-titanate-coated tube (Table 4). The relative concentrations of iron and chromium were determined (see Figure 15), and, as for the calcium-titanate coated specimen, it was found that the concentration of iron varied inversely with that of chromium. Hence, the changes which occurred in the iron-titanate-coated tubes were identical to those which occurred in the calcium-titanate-coated tube.

Quantitative point count analyses were performed at the coating-substrate interface to determine whether any titanium diffused from the coating into the substrate. As shown in Table 8, the titanium concentration was 0.06 weight percent at the surface and diminished to less than the detectability limit of 0.01 weight percent at a depth of 1.25 mils. X-ray line scans for titanium and chromium were made across the interface. These scans, shown in Figure 16, detected no significant interdiffusion of these elements.

TABLE 8
Titanium Concentrations in AISI-310 Stainless Steel Coated With Iron Titanate and Tested for 20,000 Hours at 1350°F

Depth Below Outer Surface (Mils)	Concentration (Weight Percent)
0.25	0.06
0.75	0.02
1.25	<0.01
3.75	<0.01
6.75	<0.01

Note: Total wall thickness equals 10 mils.

A full section vacuum fusion analysis of the tested specimen after the coating was removed detected 0.044 weight percent of oxygen. This represents almost no change from the as-received material which contained 0.041 weight percent of oxygen.

In summary, the iron-titanate-coated AISI-310 stainless steel tube demonstrated a stable emittance of about 0.88 for 20,000 hours at 1350°F in vacuum. Some changes in the structure of the coating occurred, but they had no observable effect on the emittance of the specimen. Coating separation occurred at the ends of the specimen and in small areas around the black-body holes, but the coating in other areas remained well bonded to the substrate. Only a very limited amount of interaction occurred between the coating and the substrate. Similar to the calcium-titanate-coated tube, substantial amounts of sigma and carbide phases precipitated in the substrate during the test. The lesser as-tested hardness increase observed in this specimen compared to the calcium titanate-coated specimen is believed due to the slight differences in microstructure commented on above.

IV. THERMAL CYCLING TESTS

A. Introduction

The two thermal cycling endurance tests of iron titanate-coated columbium-1 percent zirconium tubes were continued during the report period. One specimen was cycled from 1500 to 1800°F while the other was cycled from 1500 to 1900°F. The tests are conducted in vacuum of 1 x 10^{-7} mm Hg or better. The 96-minute cycling sequence is shown in Figure 17. During the report period the specimen cycled from 1500 to 1800°F completed 5,000 hours of exposure and the test was terminated. However, in May 1967 the contract was amended to provide for extension of the 1500 to 1900°F test to 10,000 hours.

The specimen which completed the 5,000-hour, 1500 to 1800°F test was subjected to thorough chemical and metallurgical analyses. The coating was analyzed by x-ray diffraction and spectrographic techniques while the substrate was examined for microhardness and microstructure. In addition, electron beam microprobe and vacuum fusion gas analyses were performed to detect any interaction between coating and substrate.

B. Test Results

1. 1500 to 1800°F Test

With the addition of 2100 hours of testing during this report period, the columbium-1 percent zirconium tube coated with iron titanate has completed the scheduled 5,000-hour, 1500 to 1800°F thermal cycling test. The heating and cooling time for the test was about 90 hours. The specimen was cycled a total of 3,125 times with 1,815 cycles occurring during the current report period. Of the 4910 hours remaining, 820 were at 1500°F, 3835 were at 1700°F, and 255 were at 1800°F. No adverse effects on the emittance or adherence of the coating resulted from the thermal cycling test. The appearance of the specimen at 1700°F prior to completion of the test is shown in Figure 18. The small wedge-shaped hair-line crack seen at the upper end of the specimen appeared after about 3,500 hours of testing.

The emittance of the specimen was stable at 1800°F at about 0.88 for the duration of the test as shown in Figure 19. A vacuum of 1 x 10^{-7} mm Hg or better was maintained in the test chamber (see Figure 19).

Examination of the specimen after termination of the test revealed no changes in the texture or color of the coating and no indication of coating separation or spalling. The hairline crack was hardly visible to the naked eye and is not discernible in the photograph shown in Figure 20 which was taken after completion of the test.

The impurities in the coating as detected by semi-quantitative emission spectrographic analyses are shown in Table 9.

TABLE 9

Results of Semi-Quantitative Spectrographic Analyses for Iron Titanate Coating On Columbium-1 Percent Zirconium Tube

	Impurity Content (Weight Percent)
As Sprayed	0.5%Al, 0.2%Mg, 0.4%Mn, 0.2%Cr, 0.4%Si, trace Cu
After Test	0.5%Al, 0.5%Mg, 1.0%Mn, 0.1%Cr, 0.5%Si, trace Cu

X-ray diffraction analyses showed that some changes in the structure of the coating occurred during the test. Before testing, the coating was entirely iron titanate. After testing, the phases present were iron titanate and titanium oxide in about equal amounts.

The columbium-1 percent zirconium substrate was examined metallographically in the as-received condition as well as after testing. The microstructure of the as-received material shown in Figure 21 is typical of that for cold-worked columbium-1 percent zirconium. Both etched and unetched photomicrographs of the tested specimen are shown in Figures 22 and 23 respectively. The appearance of the microstructure indicates that partial recrystallization occurred during testing.

Microhardness traverses were made on cross-sections of the as-received columbium-1 percent zirconium tube and on the tube after testing. Measurements were made with a Reichert microhardness tester at a load of 43 grams. The as-received tube had an average diamond pyramid hardness of about 145 kg/mm^2. The hardness level indicates that the material received on the order of 50 percent cold working following the last heat treatment. Four microhardness traverses were made across the wall of the tested tube. As shown in Figure 24 and Table 10, the most notable changes were a hardness gradient from the outer surface to a depth of about 4 mils in addition to an overall increase in hardness.

Electron microprobe analyses were conducted to determine if any diffusion of coating elements occurred. Quantitative point count analyses for titanium and iron were made at the coating-substrate interface. The titanium concentration

TABLE 10

Microhardness Traverse Results for Columbium-1 Percent Zirconium Tube Coated with Iron Titanate and Thermally Cycled between 1500 and 1800°F for 5,000 Hours

Traverse 1

Depth (Mils)	Diamond Pyramid Hardness (kg/mm^2)
0.5	258
2.5	231
4.0	215
6.0	215
7.5	219
8.5	219
9.5	201

Traverse 2

Depth (Mils)	Diamond Pyramid Hardness (kg/mm^2)
0.5	262
2.0	237
4.0	223
5.5	219
7.5	219
9.25	194

Traverse 3

Depth (Mils)	Diamond Pyramid Hardness (kg/mm^2)
0.5	258
2.0	249
3.0	236
4.0	213
5.75	213
7.25	213
9.25	190

Traverse 4

Depth (Mils)	Diamond Pyramid Hardness (kg/mm^2)
0.75	263
2.0	245
2.75	228
4.0	213
6.0	213
7.5	213
9.0	187

Note: Total wall thickness equals 10 mils.

decreased from 0.08 weight percent at the surface to less than 0.01 weight percent at a depth of 1.5 mils. The iron concentration measured 0.84 weight percent at the surface and decreased to less than 0.01 weight percent at a depth of 4.25 mils. The results are listed in Table 11. The amount of iron and titanium diffusion is slightly less than that which was reported after the 10,000-hour endurance test at 1700°F (see PWA-2877). X-ray line scans were made across the interface for titanium and columbium. These scans, shown in Figure 25, detected no significant interdiffusion of these elements.

TABLE 11

Concentrations of Iron and Titanium Across Columbium-1 Percent Zirconium Tube Coated with Iron Titanate and Thermally Cycled between 1500 and 1800°F for 5,000 Hours

Distance from Outer Surface (Mils)	Concentration (Weight Percent) Iron	Titanium
0.2	0.84	0.08
0.5	0.66	0.03
1.5	0.42	0
2.75	0.12	0
4.25	0	0

Note: Minimum detectable concentration for iron and titanium is 0.01 percent. Concentrations below this level are reported as zero. Total wall thickness equals 10 mils.

Vacuum fusion oxygen analyses were performed on the columbium-1 percent zirconium substrate in the as-received condition and after testing. The as-received material was found to contain about 0.03 weight percent of oxygen. Three full-section analyses were made on the tested specimen after the coating was mechanically removed and the surface cleaned by acid etching. The average of the three analyses showed the oxygen content to be 0.42 weight percent.

The hardness gradient and overall increase in hardness of the substrate can be generally accounted for by the results obtained from microprobe and vacuum fusion analyses.

First, the solid solution hardening effects of iron and titanium were considered. One weight percent of iron increases the diamond pyramid hardness by 50 kg/mm^2 over the base hardness[1] and one weight percent of titanium increases the hardness by 40 kg/mm^2. From the known concentrations of iron and titanium in the substrate, the increases in hardness due to solid solution hardening were calculated. The results are shown in Figure 26.

1 Physical and Mechanical Properties of Columbium and Columbium-Base Alloys, Defense Metals Information Center Rept. 125, Battelle Memorial Institute, Columbus, Ohio, Feb. 22, 1960

Excluding the gradient at the outer surface which is mostly accounted for by iron and titanium diffusion, the hardness of the tested substrate is about 120 to 130 kg/mm^2 higher than it would be in the fully annealed condition. The solid solution hardening effect of oxygen is 85 kg/mm^2 per 0.1 weight percent[1]. Also, it has been calculated[2] that the first 0.25 weight percent of oxygen which enters the alloy would preferentially combine with zirconium and precipitate as zirconium oxide particles. This precipitate has little effect on the room temperature hardness of the alloy[1]. Therefore, the total amount of oxygen required to produce an increase in hardness of 120 to 130 kg/mm^2 is calculated to be about 0.4 weight percent. The results of vacuum fusion oxygen analyses, therefore, support the over-all elevation in hardness which occurred as a result of testing. Probable sources of oxygen as reported earlier are residual gases in the test chamber, a thin oxide film formed on the inner surface during plasma-arc spraying, and the iron-titanate coating. Data obtained from an aging test independent of this contract of an uncoated columbium-1 percent zirconium tube has verified that residual gases in the test chamber are a source of oxygen. An uncoated tube identical to the one which was coated with iron titanate and cycled for 5,000 hours was aged for 4,000 hours at 1700°F in a vacuum of 1×10^{-7} mm Hg or better. After aging, full-section vacuum fusion analyses detected 0.18 weight percent oxygen. Before testing, the columbium-1 percent zirconium tubing contained 0.03 weight percent oxygen. Consequenlty, a significant portion of the oxygen detected in the substrate of the iron-titanate coated specimen after the 5,000-hour cycling test came from residual gases in the test chamber. However, since the coated tube was cycled between 1500 and 1800°F and the tests differed in duration, the exact amount of oxygen detected in the substrate of the coated tube attributable to residual gases cannot be calculated.

In summary, the iron titanate-coated columbium-1 percent zirconium tube which was cycled 3,125 times from 1500 to 1800°F in vacuum had a stable emittance of about 0.88 and demonstrated excellent coating-to-substrate adherence during the 5,000-hour test. Some changes occurred in the coating during testing but no adverse effects on the emittance or adherence resulted. Examination of the substrate after testing revealed partial recrystallization to have occurred. Also, an overall increase in hardness and a hardness gradient at the outer surface were detected. Limited diffusion of iron and titanium occurred across the coating-substrate interface. A significant amount of oxygen was detected in the substrate and oxygen probably diffused across both the inner and outer surfaces. The

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[1] Physical and Mechanical Properties of Columbium and Columbium-Base Alloys, Defense Metals Information Center Rept. 125, Battelle Memorial Institute, Columbus, Ohio, Feb. 22, 1960

[2] Walek, W. J. and W. L. Luoma, Semi-Annual Progress Report on Determination of the Emissivity of Materials, PWA-2877, NASA CR-72058, Pratt & Whitney Aircraft, Contract NAS3-4174, June 30, 1966

hardness gradient at the outer surface and the overall increase in hardness were attributed to the solution hardening effects of iron, titanium and oxygen.

2. 1500 to 1900°F Test

With the addition of 3,560 hours of testing during this report period, an iron titanate-coated columbium-1 percent zirconium tube has completed 5,000 hours of 1500 to 1900°F thermal cycling testing. The specimen has been cycled a total of 3,115 times with 2,215 cycles occurring during this period. The test will be continued to 10,000 hours.

The emittance has remained stable throughout the test with values measuring about 0.89. The emittance values and the vacuum maintained in the test chamber as a function of time are shown in Figure 27.

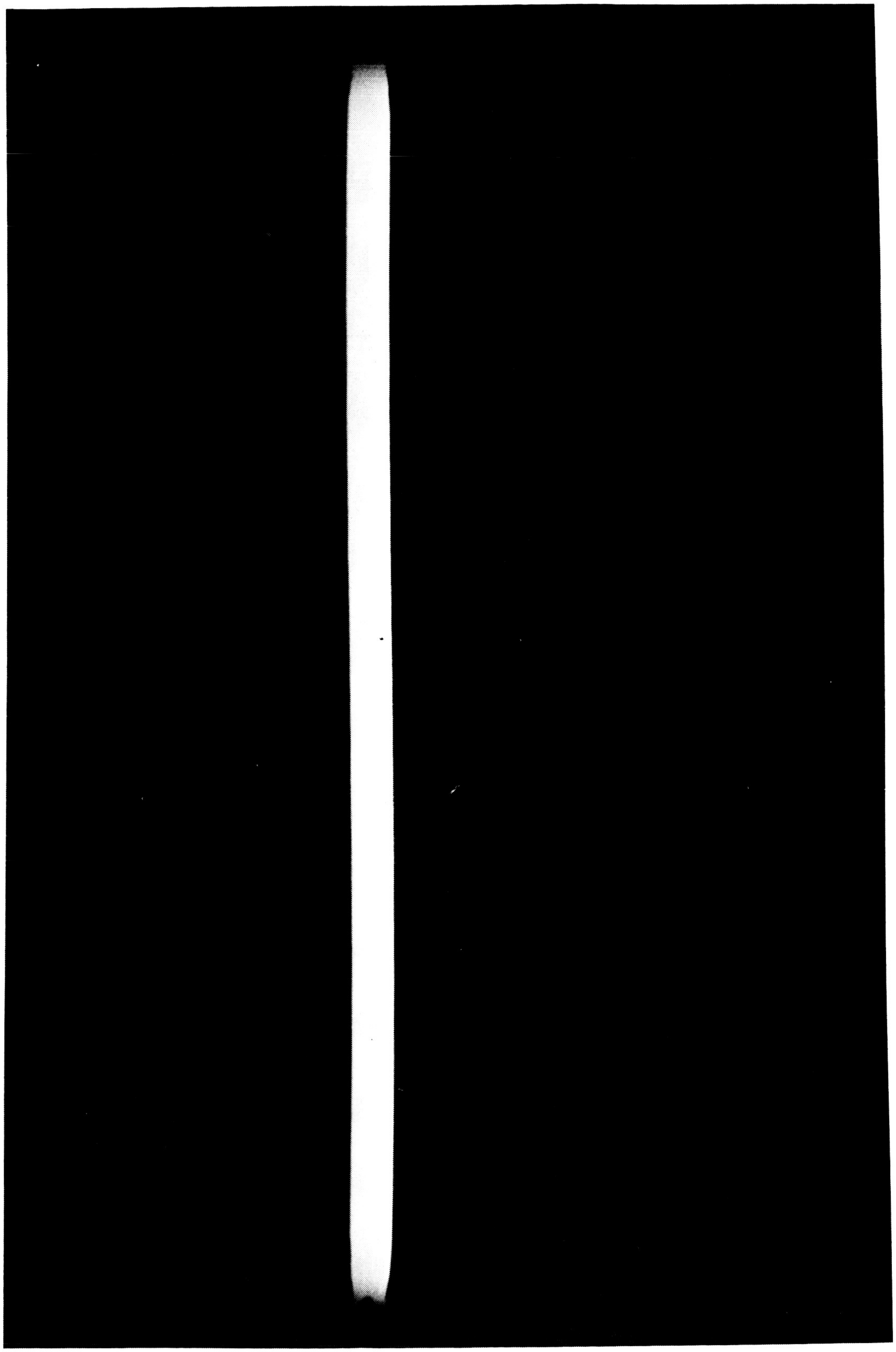
After about 2800 hours of testing, hairline cracks were observed at the ends of the specimen. These cracks occurred in the areas of the tube which were crimped after spraying to permit electrical connections to be made. After about 4900 hours the cracking extended towards the center of the tube, although the intensity of the additional hairline cracks was lower relative to those which developed earlier. Figure 28 shows the specimen at 1500, 1700 and 1900°F after about 5,000 hours of testing. However, despite the cracking, no spalling of the coating occurred, which indicates a good coating-to-substrate bond. Since there was no loss of coating-substrate contiguity, the emittance of the specimen was not significantly affected and the specimen continued to perform satisfactorily.

V. CONCLUSIONS

A. On the basis of long-term emittance testing and post-test evaluation of the calcium titanate-coated AISI 310 stainless steel tube, calcium titanate is a suitable coating for space radiator use at 1350°F. The coating demonstrated a stable emittance of 0.89 and had excellent adherence for the duration of the 20,000-hour test. No changes in the structure of the coating occurred and no significant interaction between the coating and substrate was detected. Significant changes did occur in the Type 310 stainless steel. Substantial amounts of sigma and carbide precipitates formed in the austenitic matrix. These changes can be expected in Type 310 stainless steel under the conditions of test and are not considered to be a function of the coating.

B. Long-term testing in vacuum has shown that iron titanate coatings applied to AISI 310 stainless steel have a stable emittance of about 0.88 at 1350°F for up to 20,000 hours. Some changes in the structure of the coating occurred during testing but these changes had no adverse effects on the emittance. Also, some coating separation occurred at the ends of the specimen and in small areas around the black-body holes. Only limited interraction between the coating and substrate was detected. As in the case of the calcium titanate-coated tube, substantial amounts of sigma and carbide phases precipitated throughout the substrate during the test. In view of the coating separation which occurred in the iron titanate-coated specimen, calcium titanate is considered to be a more suitable coating for application to Type 310 stainless steel.

C. The results of a 5,000-hour cycling test between 1500 and 1800°F of an iron titanate-coated columbium-1 percent zirconium tube indicates that this coating is suitable for space radiator application up to 1800°F. The coating had a stable emittance of about 0.88 and demonstrated good coating-to-substrate adherence throughout the test, which included 3,125 cycles. Some changes in the coating structure occurred but these changes had no adverse effects on the emittance or adherence. Limited diffusion of iron and titanium occurred across the coating-substrate interface. The oxygen content of the substrate increased significantly, probably diffusing across both the inner and outer surfaces of the tube. The diffusion of iron, titanium, and oxygen into the substrate raised the hardness significantly above what would be expected for the annealed alloy. Except for the increase in hardness of the substrate, the diffusion apparently had no other effects on the specimen.



Mag: 0.9X

Figure 1 Appearance of Calcium-Titanate-Coated AISI-310 Stainless Steel Tube at 1350°F After 20,000 Hours of Testing X-23808

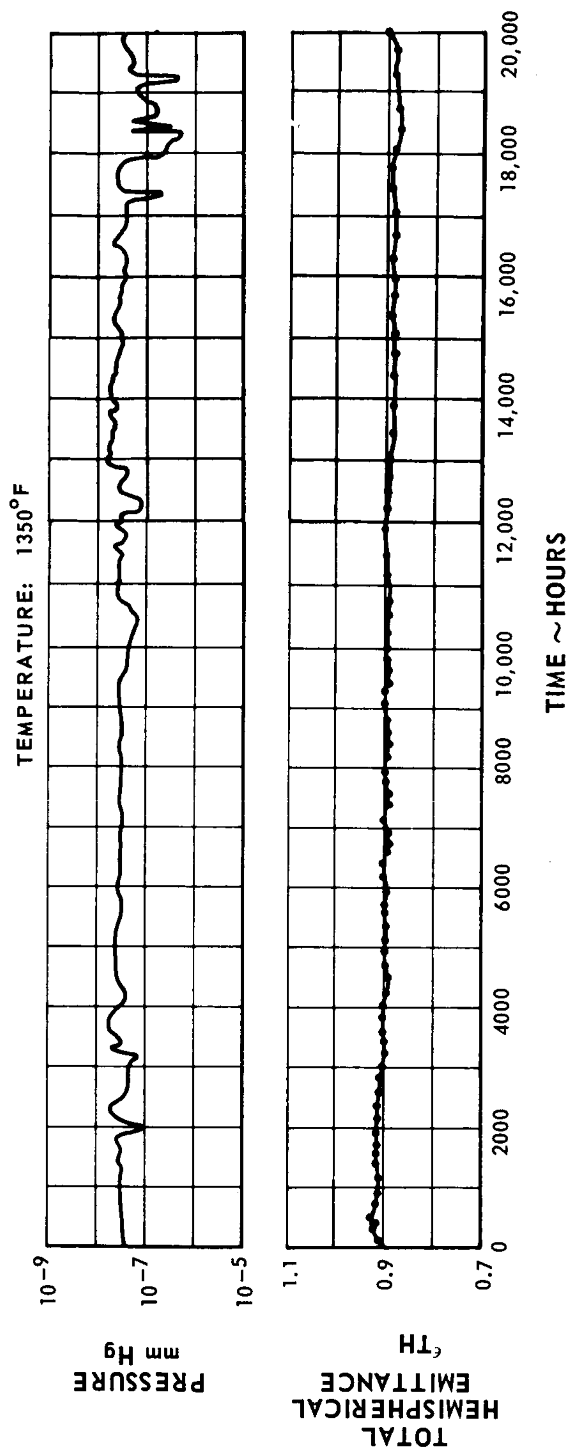
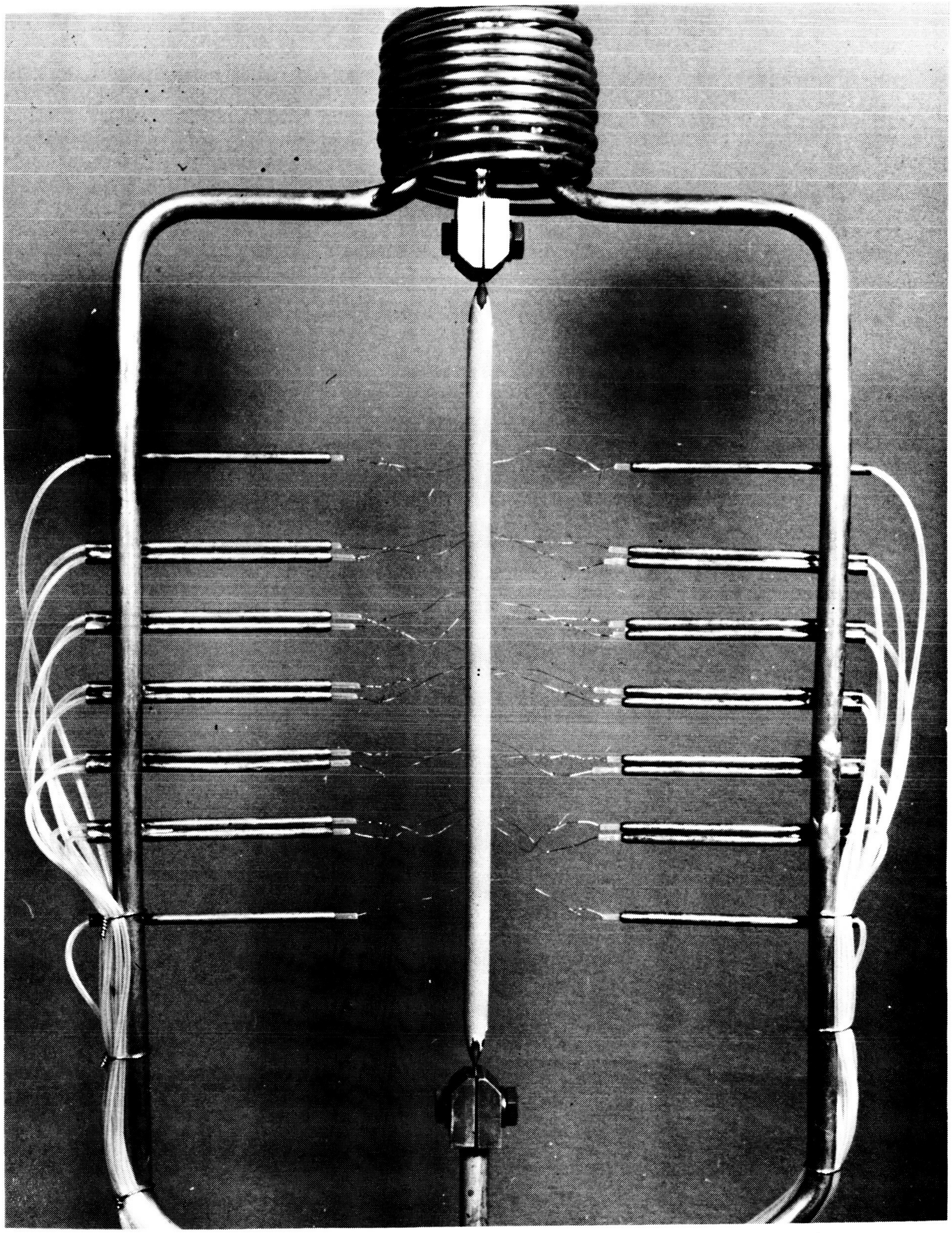


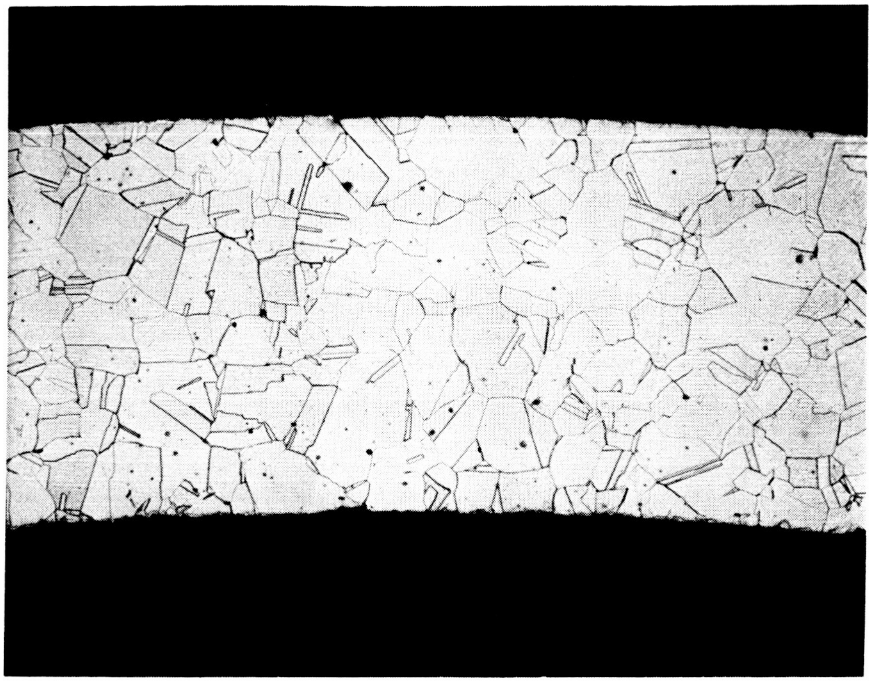
Figure 2 Pressure and Total Hemispherical Emittance for Long-Term Endurance Test of Calcium-Titanate-Coated AISI-310 Stainless Steel



Mag: 0.6X

Figure 3 Appearance of Calcium-Titanate-Coated AISI-310 Stainless Steel Tube at Room Temperature After 20,000 Hours of Testing

X-23949



Etchant: 43% Glycerine, 43% HCl, 14% HNO_3 Mag: 200X

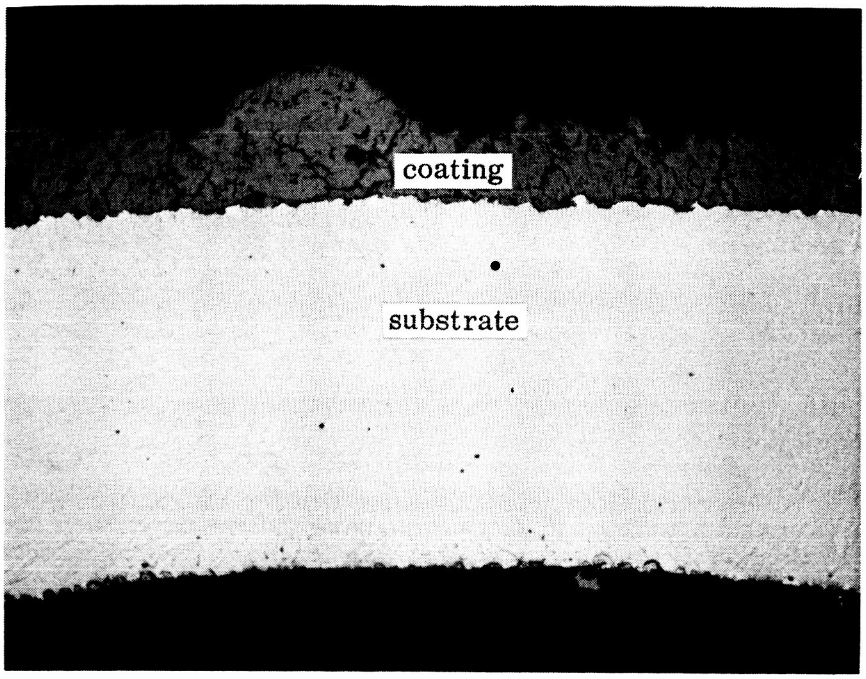
Figure 4 Photomicrograph of AISI-310 Stainless Steel Tube in the As-Received Condition

Mag: 200 X

Mag: 500X

Etchant: 43% Glycerine, 43% HCl, 14% HNO_3

Figure 5 Photomicrographs of AISI-310 Stainless Steel Tube Coated with Calcium Titanate and Tested for 20, 000 Hours at 1350°F



Mag: 200X

Figure 6 Unetched Photomicrograph of AISI-310 Stainless Steel Tube Coated with Calcium Titanate and Tested for 20,000 Hours at 1350°F

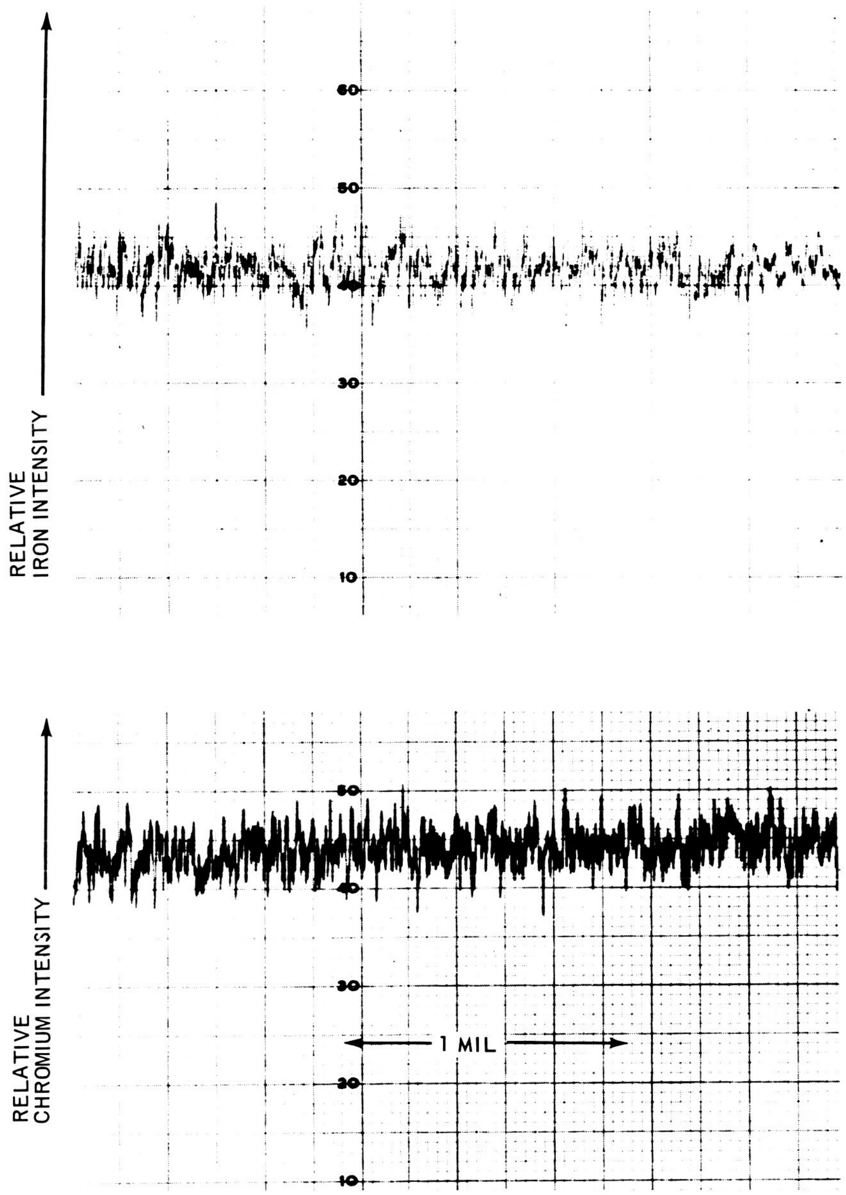


Figure 7 Relative Concentrations of Iron and Chromium in AISI-310 Stainless Steel Specimen in As-Received Condition

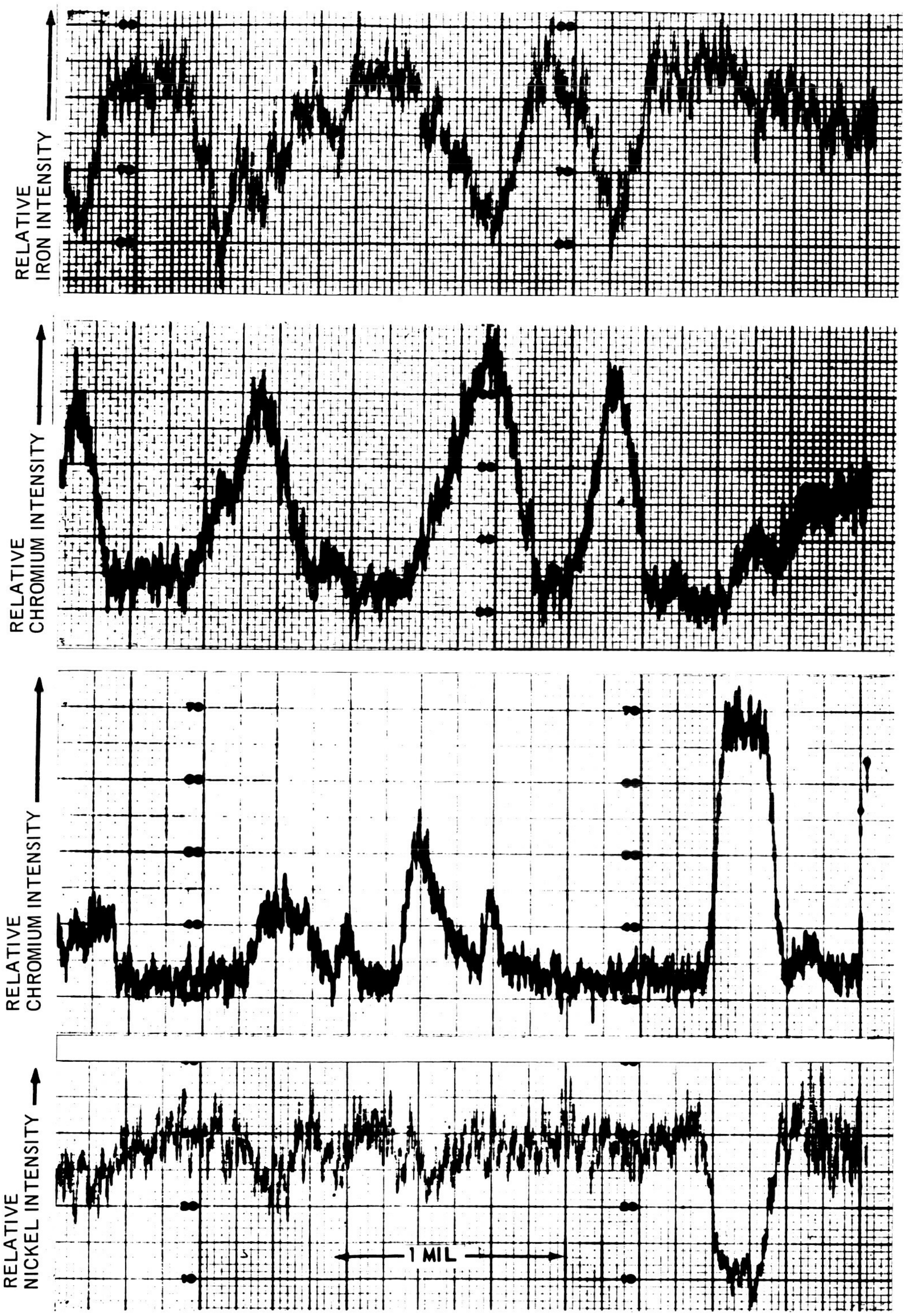
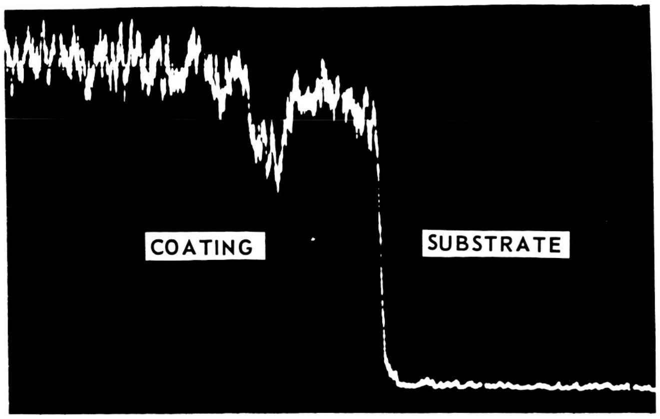
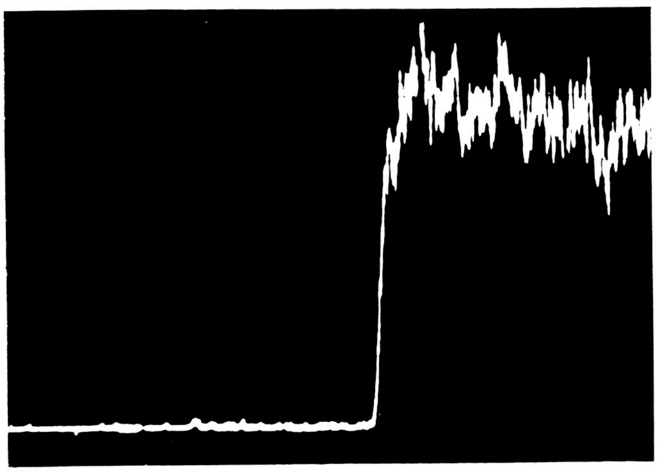


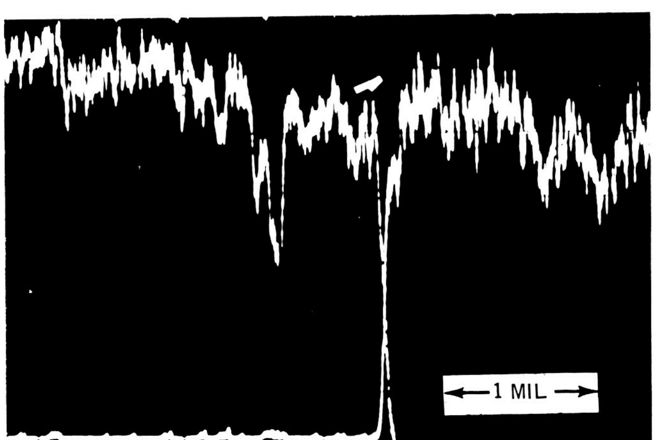
Figure 8 Relative Concentrations of Iron and Chromium and of Nickel and Chromium in AISI-310 Stainless Steel Specimen Coated with Calcium Titanate and Tested for 20,000 Hours at 1350°F



Relative Titanium Concentration



Relative Iron Concentration



Relative Composite Titanium and Iron Concentrations

Figure 9 Relative Concentrations of Iron and Titanium Across Interface of Calcium Titanate and AISI-310 Stainless Steel After Testing for 20,000 Hours at 1350°F

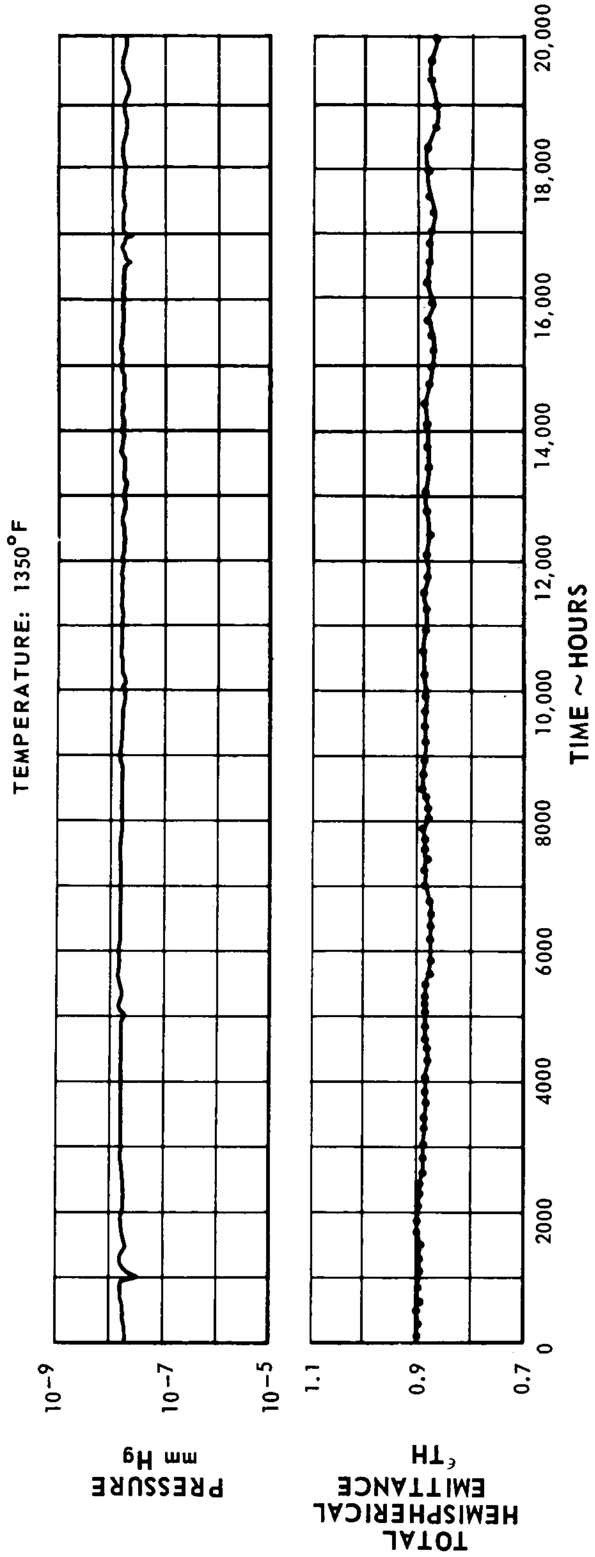
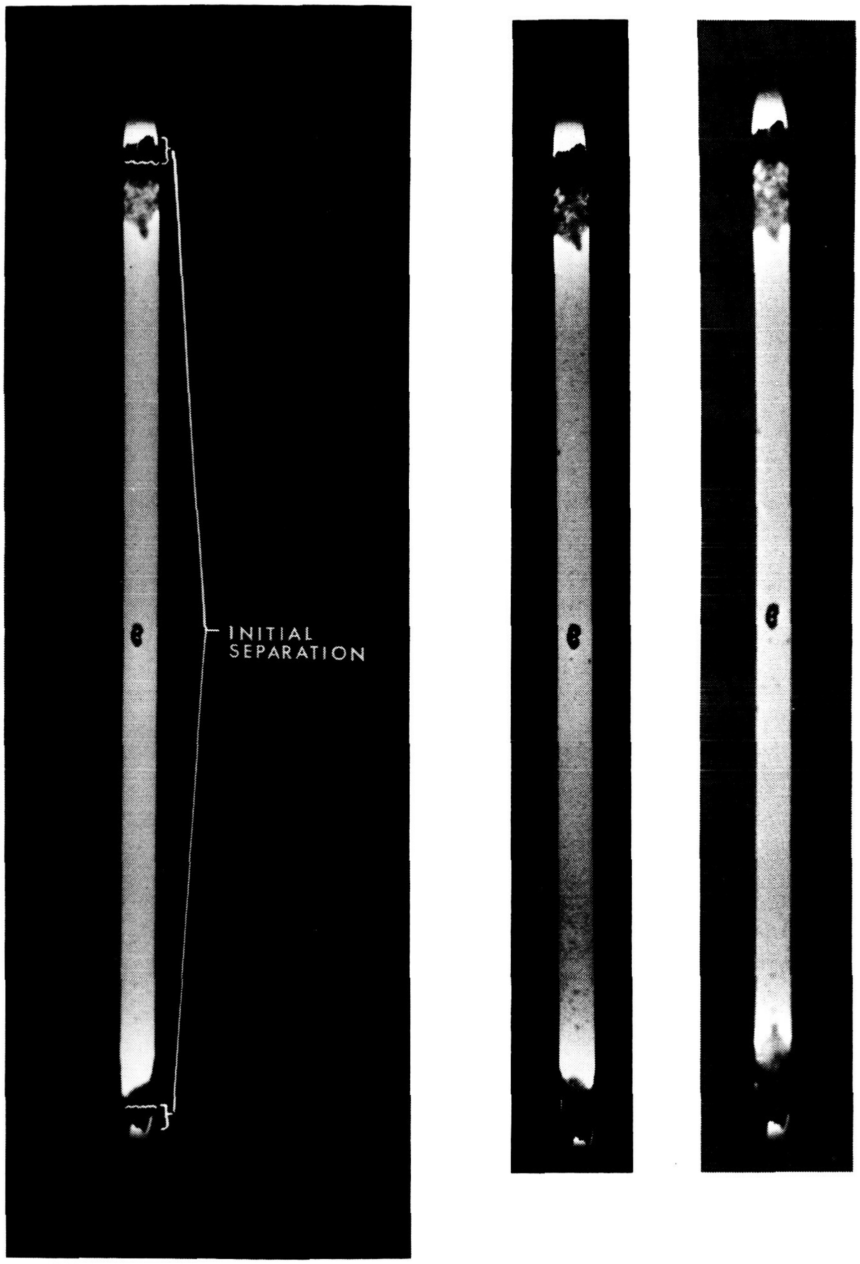


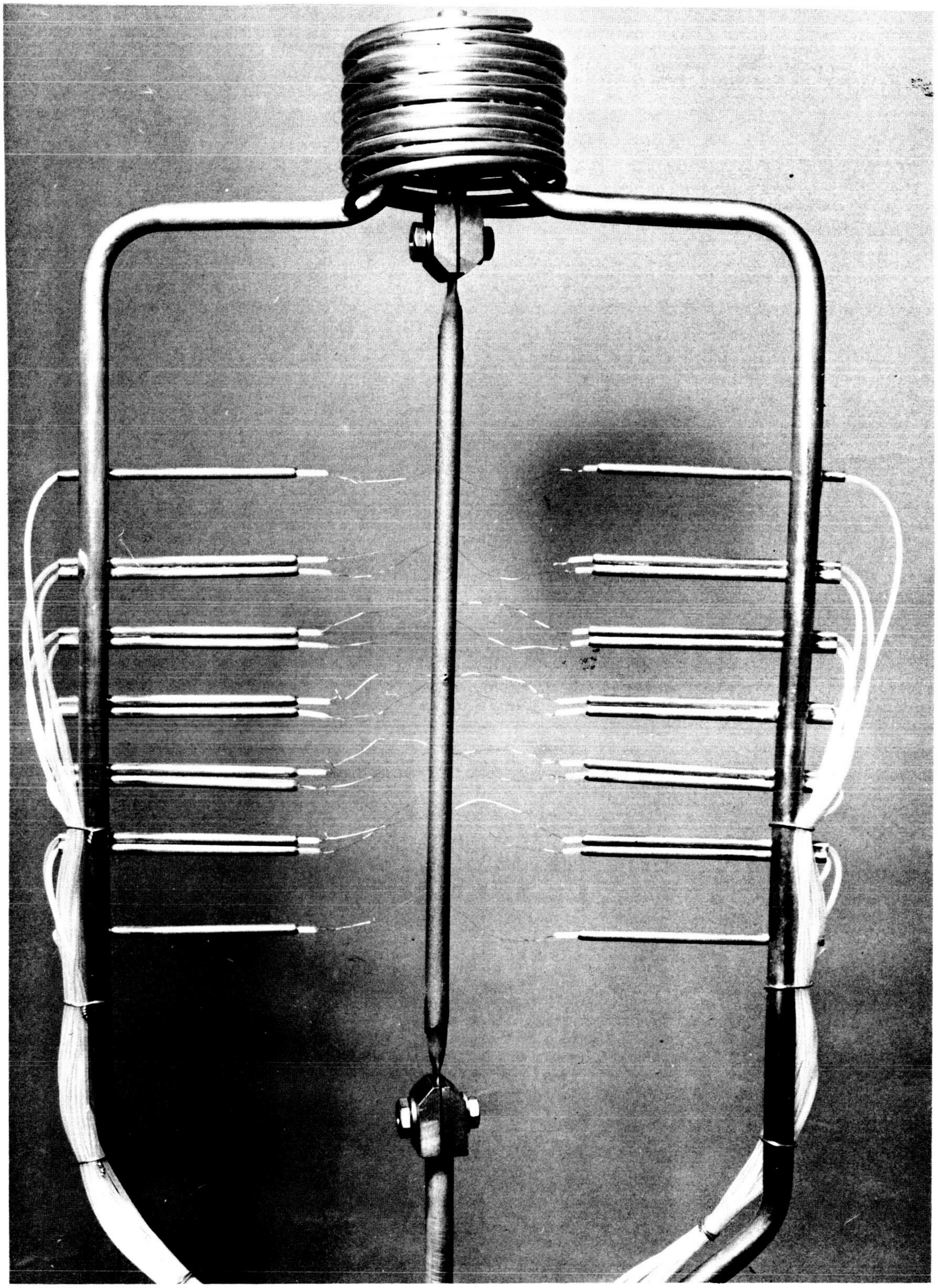
Figure 10 Pressure and Total Hemispherical Emittance for Long-Term Endurance Test of Iron-Titanate-Coated AISI-310 Stainless Steel



Mag: 0.75X

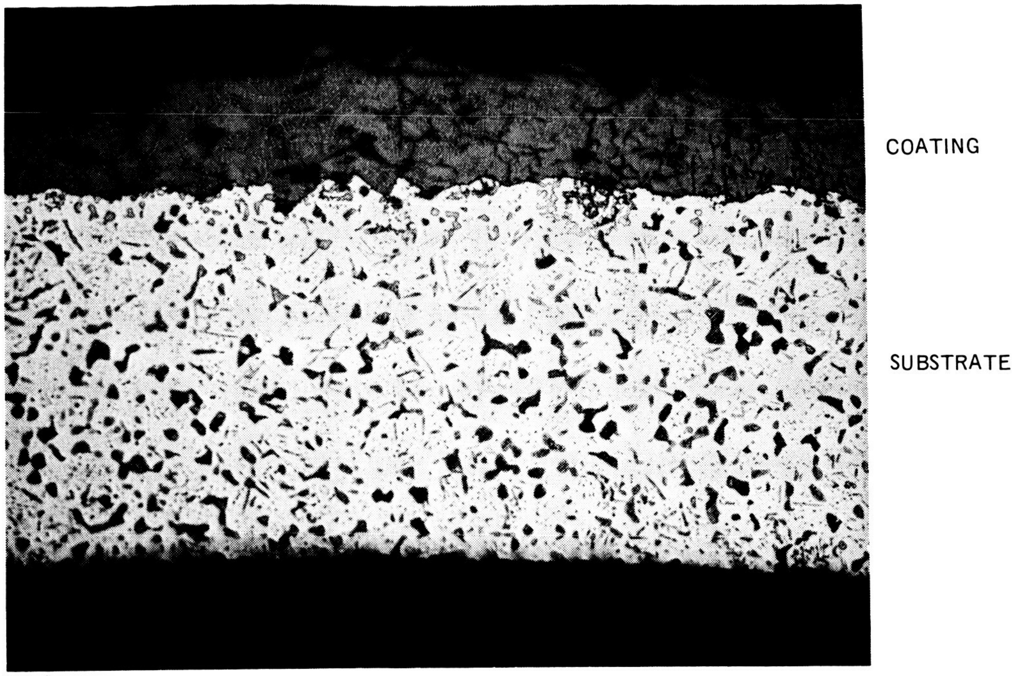
Figure 11 Appearance of Iron-Titanate-Coated AISI-310 Stainless Steel Tube at 1350°F After 15,400 Hours (Left), 17,500 Hours (Center), and 20,000 Hours (Right) Showing Progression of Coating Separation

XP-69336/XP-22281/X-24143



Mag: 0.6X

Figure 12 Appearance of Iron-Titanate Coated AISI-310 Stainless-Steel Tube at Room Temperature After 20,000 Hours of Testing X-24139

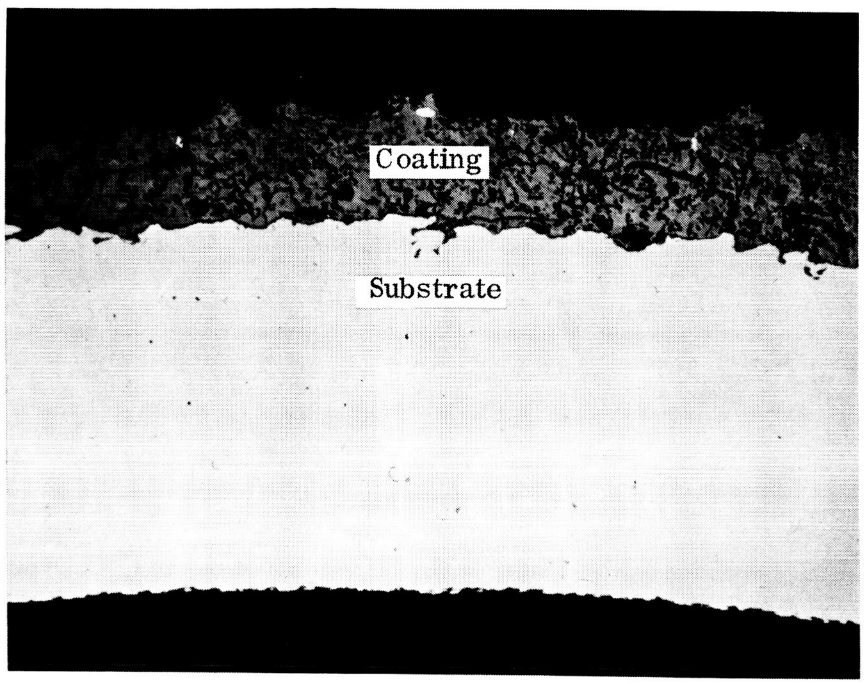


Mag: 200 X

Mag: 500X

Etchant: 43% Glycerine, 43% HCl, 14% HNO_3

Figure 13 Photomicrographs of AISI-310 Stainless Steel Tube Coated with Iron Titanate and Tested for 20, 000 Hours at 1350°F



Mag: 200X

Figure 14 Unetched Photomicrograph of AISI-310 Stainless Steel Tube Coated With Iron Titanate and Tested for 20,000 Hours at 1350°F

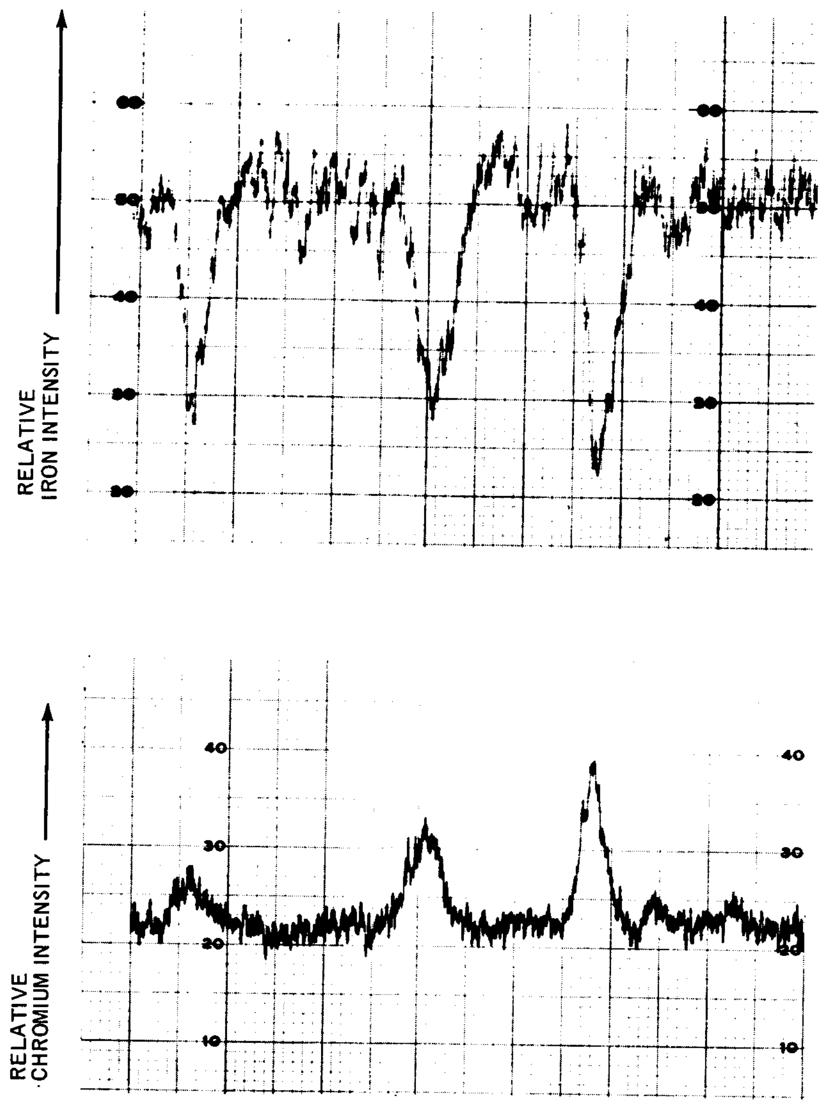
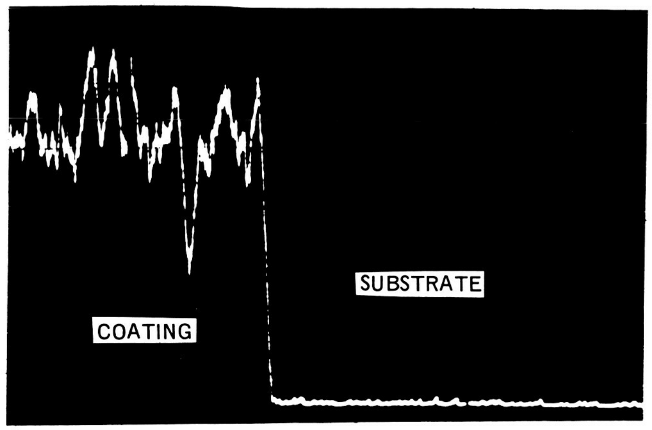
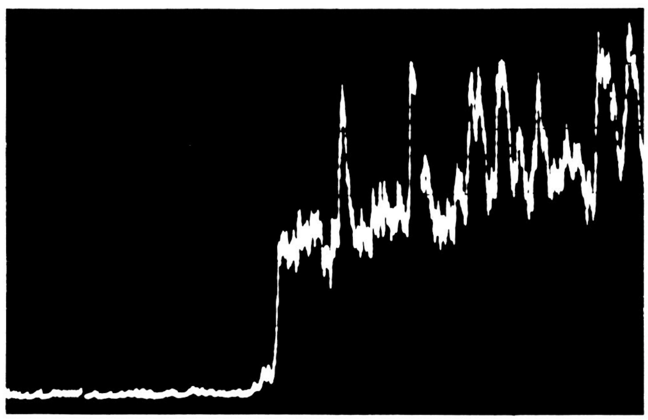


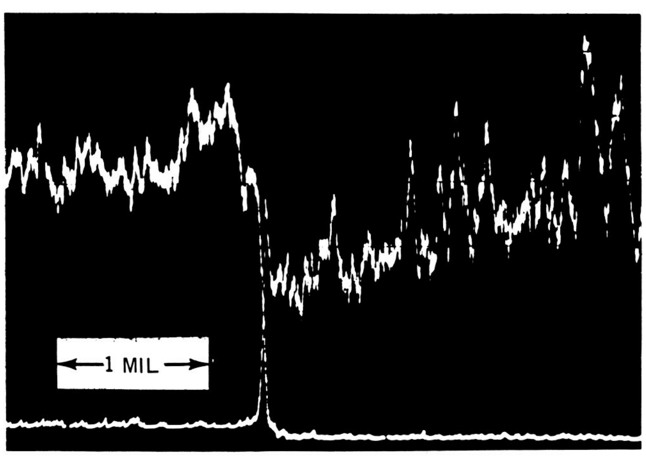
Figure 15 Relative Concentrations of Iron and Chromium in AISI-310 Stainless Steel Specimen Coated with Iron-Titanate and Tested for 20,000 Hours at 1350°F



Relative Titanium Concentration



Relative Chromium Concentration



Relative Composite Titanium and Chromium Concentrations

Figure 16 Relative Concentrations of Iron and Titanium Across Interface of Iron Titanate and AISI-310 Stainless Steel After Testing for 20,000 Hours at 1350°F

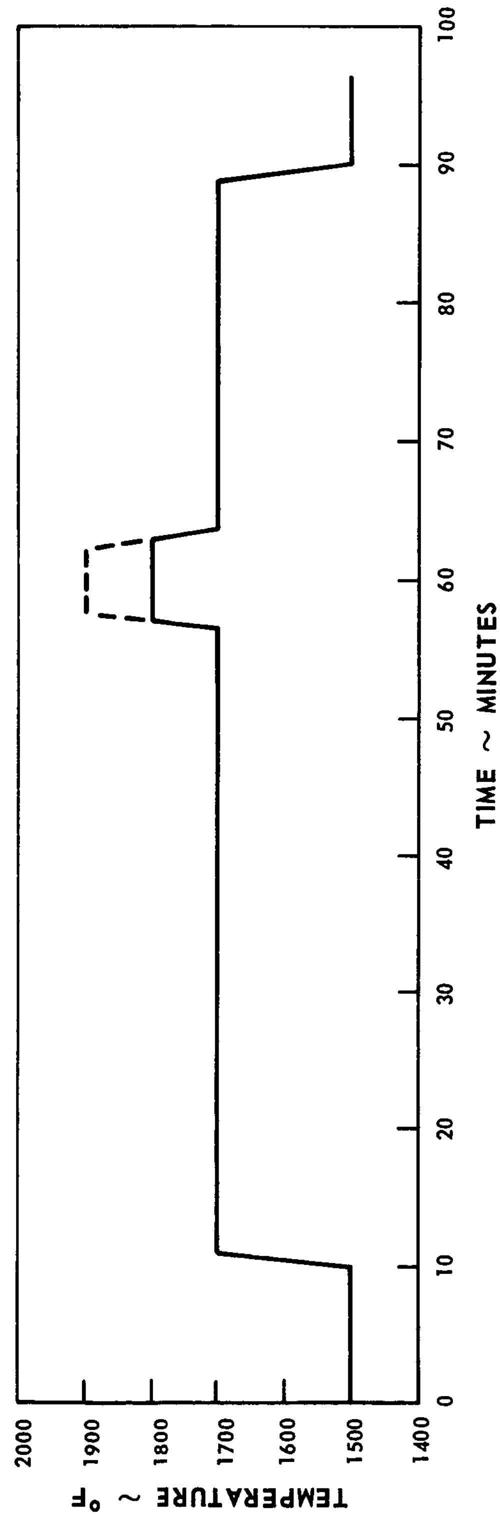
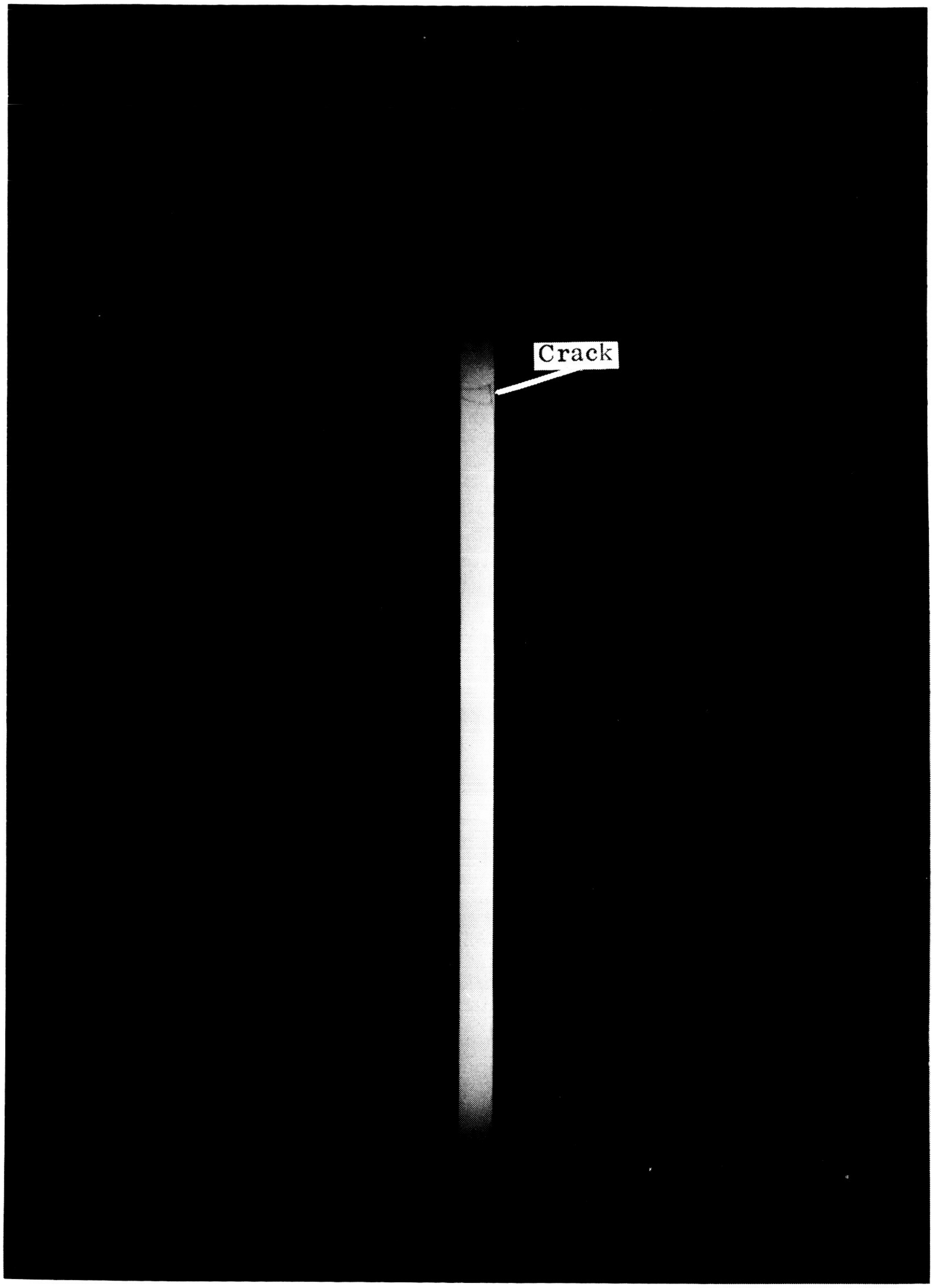


Figure 17 Thermal Cycling Sequence



Mag: 0.7X

Figure 18 Iron-Titanate-Coated Columbium-1 Percent Zirconium Tube at 1700°F in Vacuum Prior to Completion of 5,000-Hour 1500 to 1800°F Cycling Test X-24554

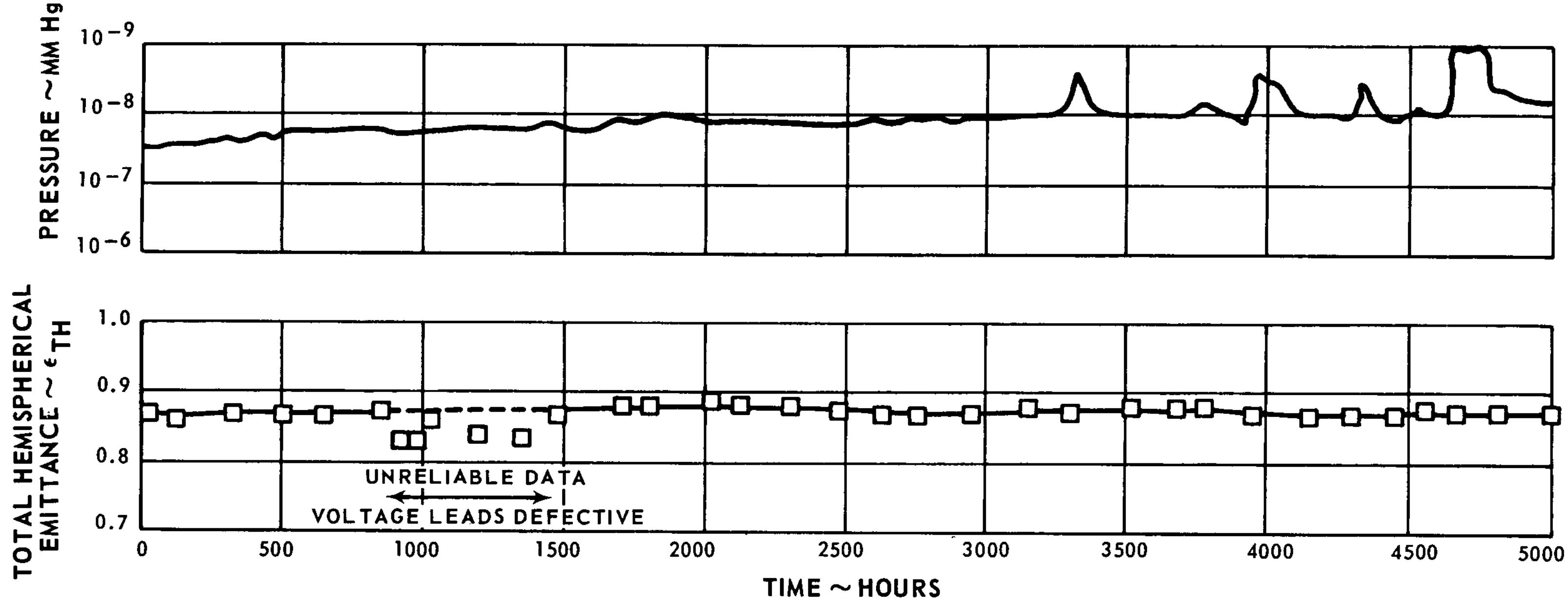
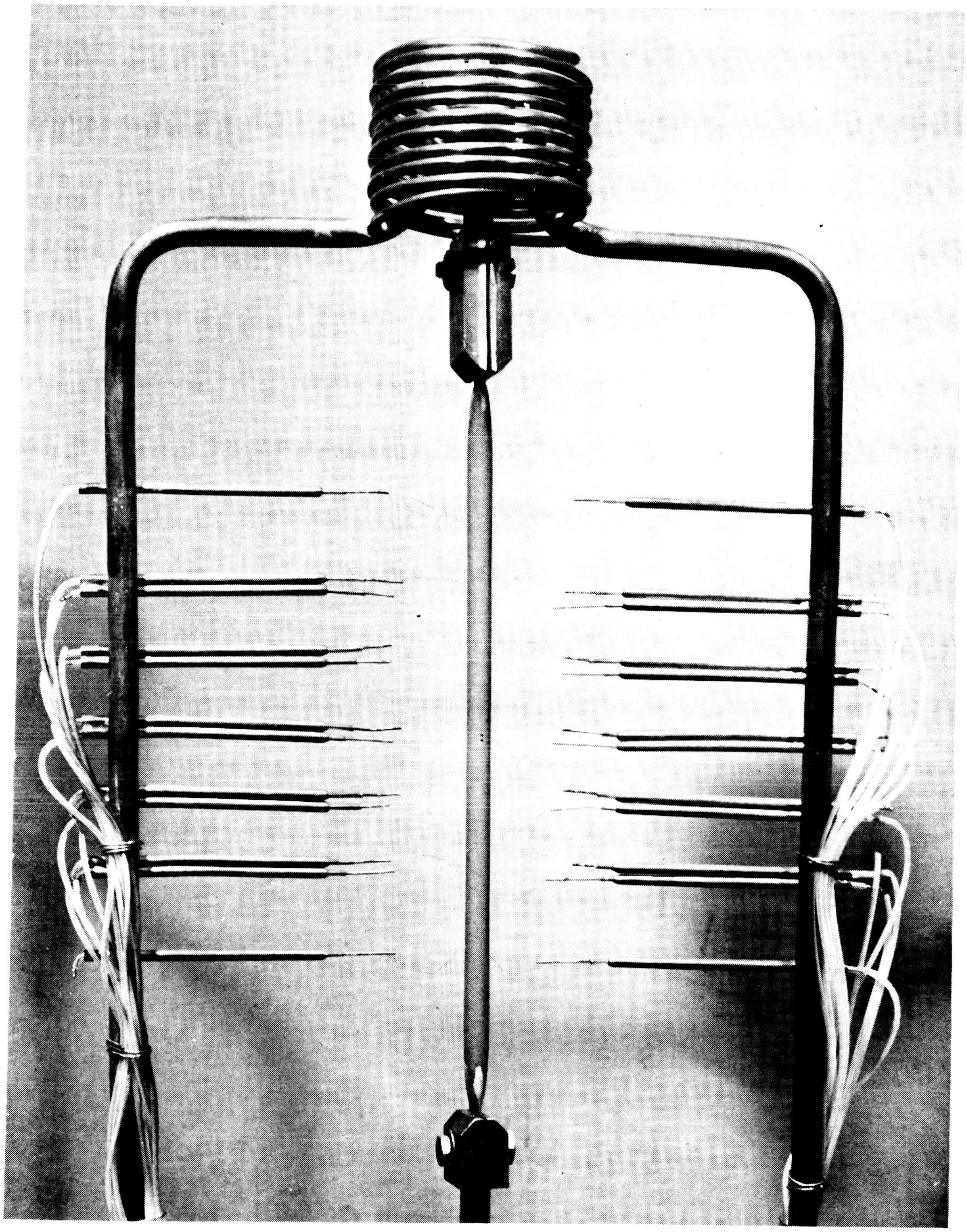


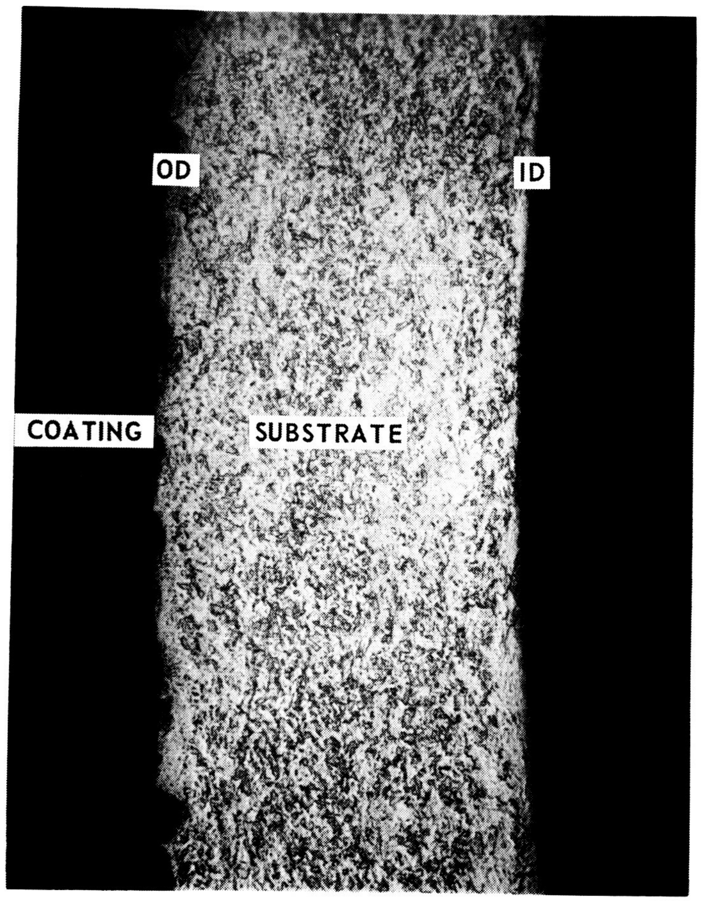
Figure 19 Pressure and Total Hemispherical Emittance for Iron Titanate-Coated Columbium-1 Percent Zirconium Tube Cycled between 1500 and 1800°F



Mag: 0.6X

Figure 20 Appearance of Iron-Titanate-Coated Columbium-1 Percent Zirconium Tube Thermally Cycled for 5,000 Hours Between 1500 and 1800°F

X-24557

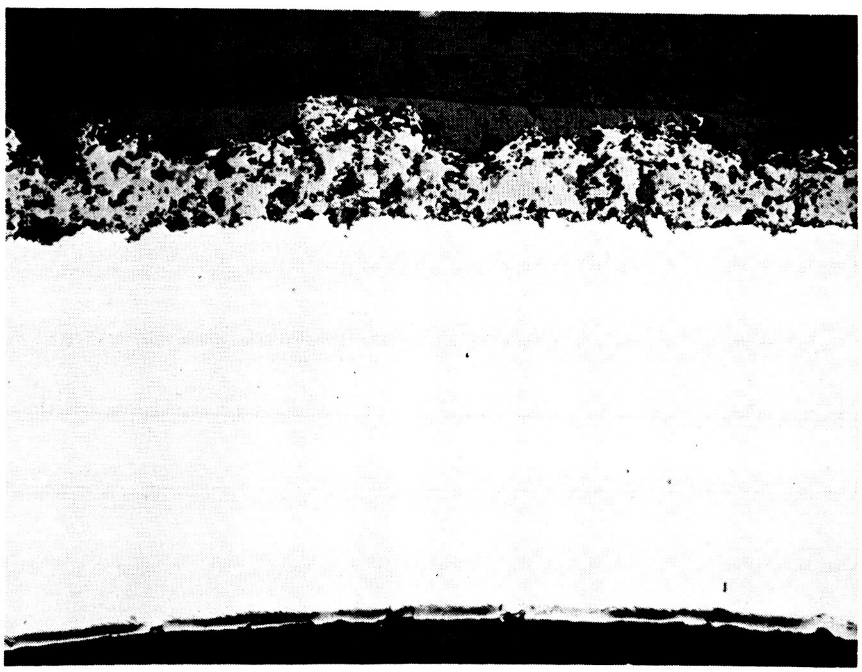


Etchant: 10% HF, 30% HNO, 60%H_2O Mag: **200x**

Figure 21 Cross Sectional Photomicrograph of Columbium-1 Percent Zirconium Tube After Being Coated With Iron Titanate

Etchant: 10% HF, 30% HNO_3, 60% H_2O Mag: 200X

Figure 22 Photomicrograph of Columbium-1 Percent Zirconium Tube Coated with Iron Titanate after Cycling between 1500 and 1800°F for 5,000 Hours



Mag: 200X

Figure 23 Unetched Photomicrograph of Columbium-1 Percent Zirconium Tube Coated with Iron Titanate after Cycling between 1500 and 1800°F for 5,000 Hours

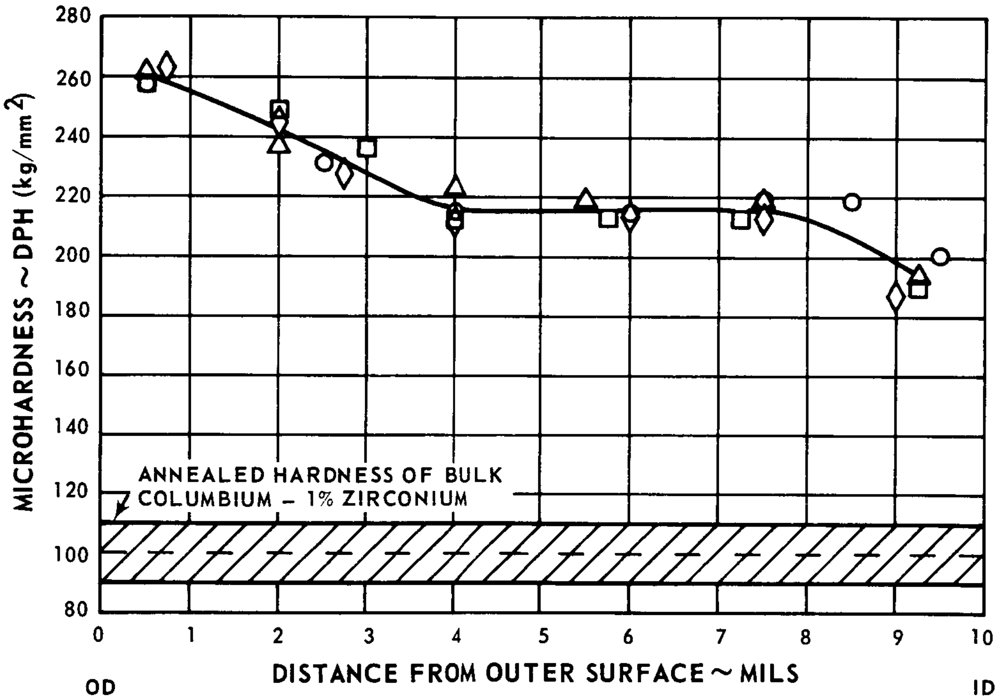
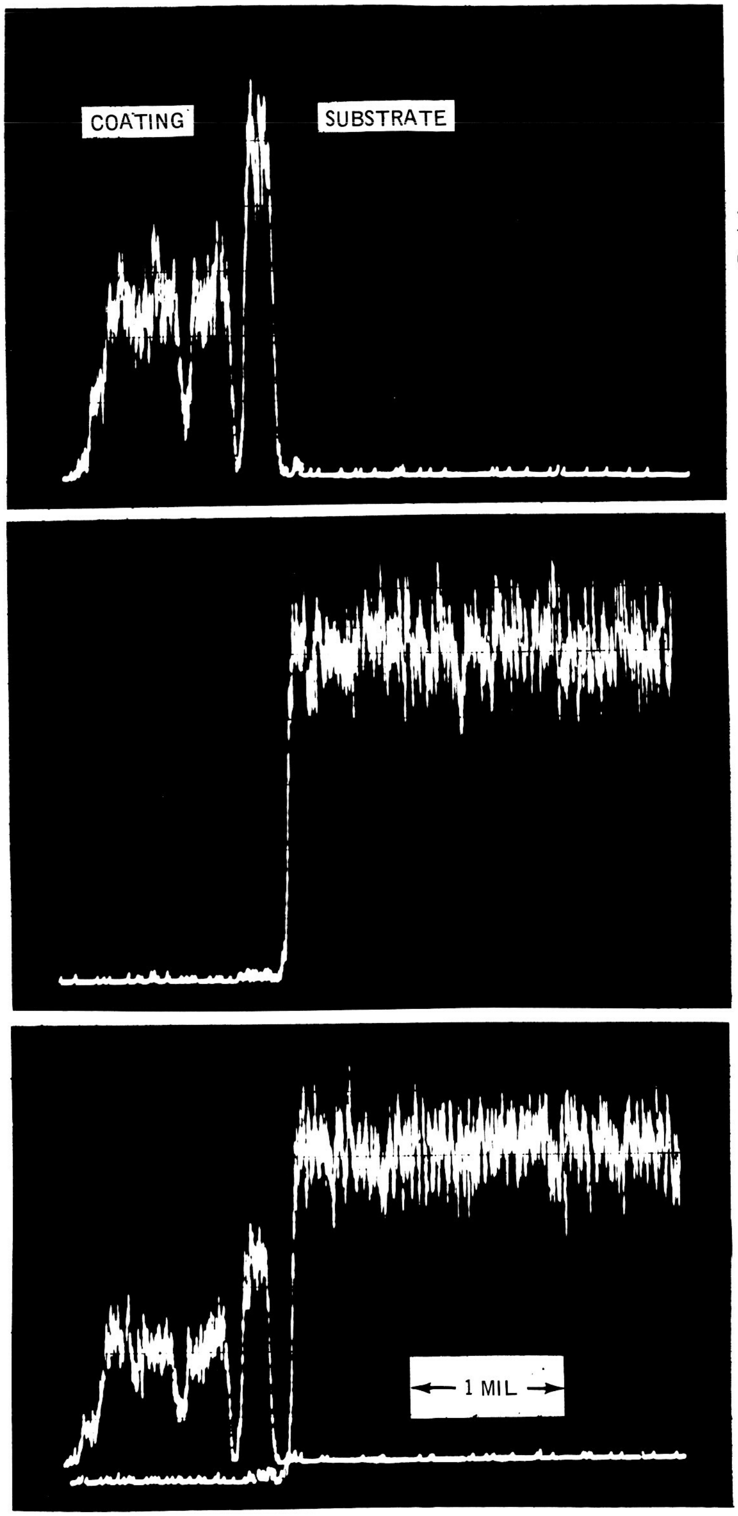


Figure 24 Microhardness Traverse of Columbium-1 Percent Zirconium Tube Coated with Iron Titanate and Thermally Cycled for 5,000 Hours between 1500 and 1800°F



Relative Titanium Concentration

Relative Columbium Concentration

Composite Concentrations of Titanium and Columbium

Mag: 800X

Figure 25 Relative Concentrations of Titanium and Columbium across Interface of Iron Titanate and Columbium- 1 Percent Zirconium after 5,000 Hours of 1500 to 1800°F Thermal Cycling Test

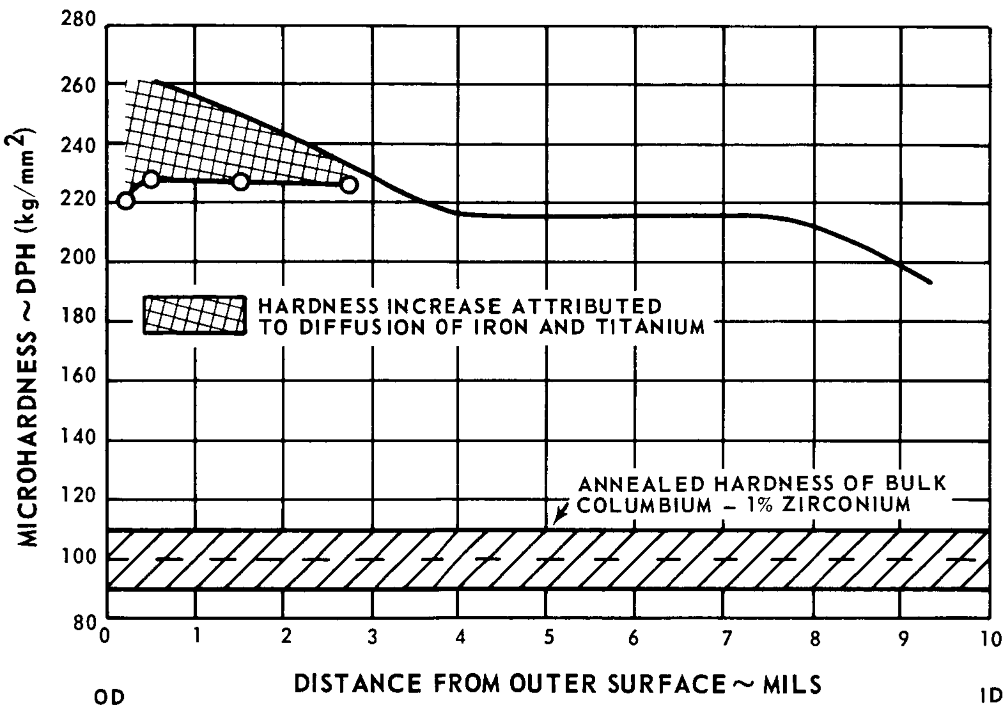


Figure 26 Effect of Iron and Titanium on Hardness of Columbium-1 Percent Zirconium Tube Coated with Iron Titanate and Thermally Cycled for 5,000 Hours between 1500 and 1800°F

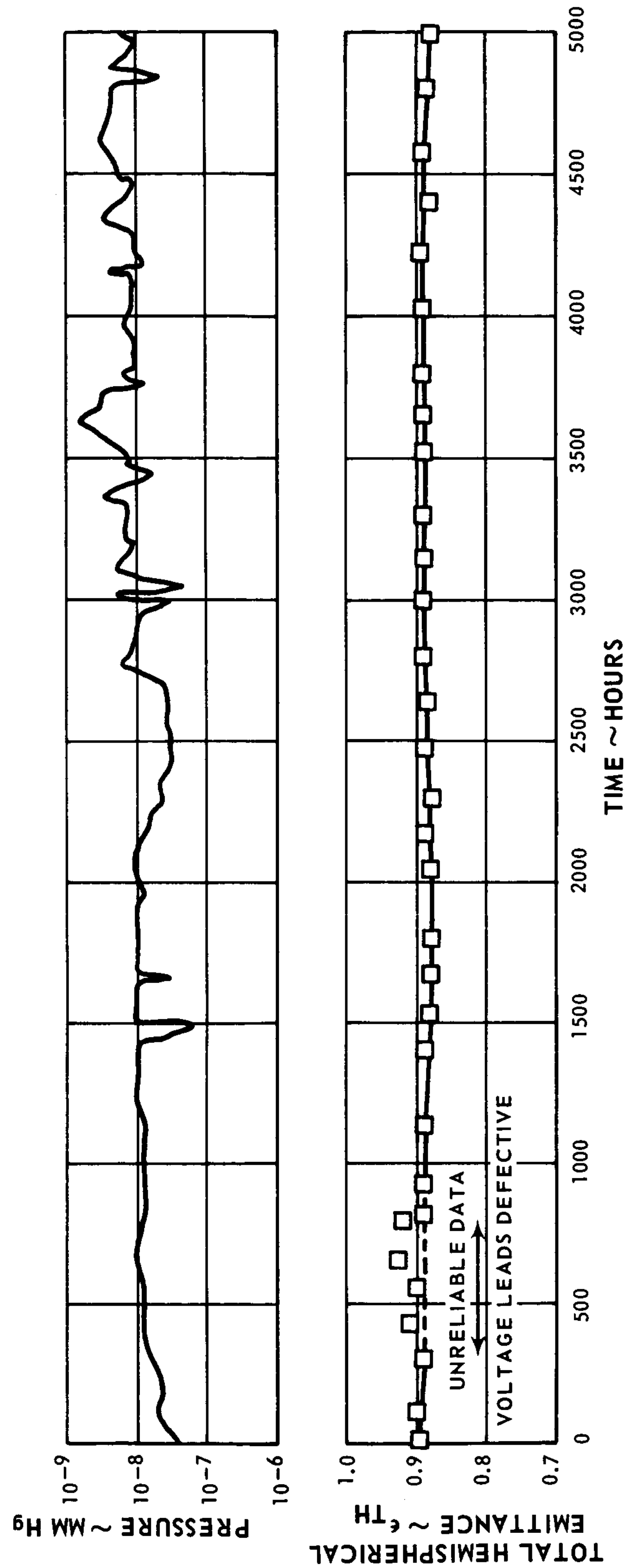
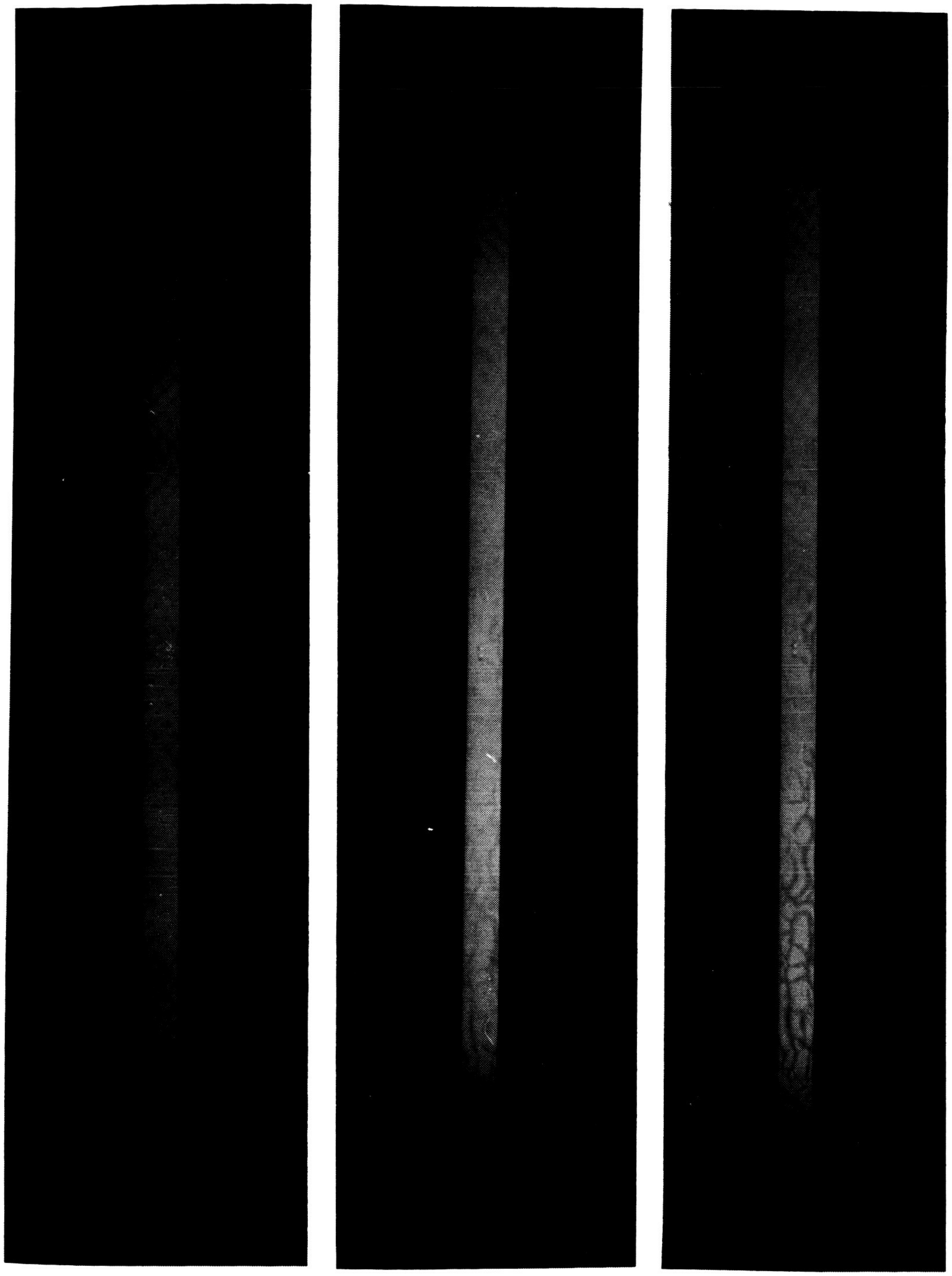


Figure 27 Pressure and Total Hemispherical Emittance for Iron Titanate-Coated Columbium-1 Percent Zirconium Tube Cycled between 1500 and 1900°F



1500°F 1700°F 1900°F

Mag: 0.7X

Figure 28 Appearance of Iron Titanate-Coated Columbium-1 Percent Zirconium Tube at 1500°F (left), 1700°F (center), and 1900°F (right) after 5,000 Hours of Testing between 1500 and 1900°F

X-24798/X-24799/X-24800

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